VOLUME 11 • NUMBER 2 • FEBRUARY, 1940

# Metals and Alloys

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Published Monthly by Reinhold Publishing Corporation, East Stroudsburg, Pa., U.S.A. Ralph Reinhold, President and Treasurer; H. Burton Lowe, Vice President and Secretary; Philip H. Hubbard, Vice President; Francis M. Turner, Vice President. Executive and Editorial Offices, 330 West 42nd Street, New York. Price 25 cents a copy. Annual Subscription: U. S., Possessions and Canada, $2.00. All Other Countries, $3.00. (Remit by New York Draft). Copyright, 1940, by Reinhold Publishing Corporation. All rights reserved. Entered as second class matter June 12, 1934, at the Post Office at East Stroudsburg, Pa., under the Act of March 3, 1879.

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# Magnesium Alloys for Photoengraving

BY ROBERT T. WOOD

*Metallurgist, American Magnesium Corp., Cleveland*

*Some months ago our attention was called to a new metal plate for photoengravers. Learning that it was a magnesium alloy plate, we approached our friends in the American Magnesium Corp., and solicited an article. We are glad to present this contribution which is characterized by our Advisory Board as a "nice article—off the beaten track and impartially written."*

*The author discusses the composition of the alloy, the preparation of the plate for etching, the etching technique, the repair of damaged plates, their life and some of the advantages or benefits that would result from their use.—The Editors.*

SOME TIME AGO, IN THESE PAGES, B. W. Gonser and S. Epstein presented a survey of the metals and alloys used in the printing industry.[1] One of the processes described was the reproduction of line etchings and half tones by the use of metal plates. The use of zinc, copper and brass in this process is well known. The purpose of this article is to summarize recent experiments to determine the possibilities of magnesium alloys for photoengravers' plates, or etching plates as they are sometimes called.

Magnesium alloy etching plates in small sizes called "Klichees" have been used in Germany for at least 10 yrs. and during the last 2 or 3 yrs. their use has greatly increased. Samples of recent work done with magnesium alloy etching plates in Germany prove that, when the proper technique is employed, the results have a high order of artistic merit. Halftone prints from magnesium alloy plates appear to have more "life" than similar zinc plates and some competent judges have asserted that for certain types of work magnesium alloy plates make more "brilliant" reproductions than copper plates. Magnesium alloy plates seem to be especially effective where sharp contrast in tone is desired.

Early attempts to introduce imported magnesium plates for photoengravers' use in this country were unsuccessful for a variety of reasons. The work here reported was undertaken by the American Magnesium Corp. to determine whether photoengravers' plates constituted a sound application for magnesium. A preliminary survey revealed that the problem was not easy either from the metallurgical or economic viewpoint. An entirely unknown factor was the reaction of the photoengravers to a new metal entailing changes in established practice.

## Sheet Manufacture—Alloy Composition

The quality of sheet required by photoengravers is so high that zinc and copper plates are manufactured in plants that specialize in this type of work. Ordinary high grade commercial sheet is unacceptable because minute pits, streaks or scratches which would not be considered objectionable for any ordinary use must be entirely absent from photoengravers' plate for any of these would probably cause trouble during etching.[1] In addition, photoengravers' sheet must be free from hard spots, have a uniform small grain size, be of uniform temper and extremely flat.

A few years ago, it would have been considered impossible to produce 22 by 28-in. plates from magnesium alloy sheet to meet these requirements on a commercial basis but the technology of fabricating magnesium alloys has advanced so rapidly that this problem has been satisfactorily solved. In common with other metallurgical products, the quality of the finished sheet depends upon the ingot from which it is rolled.

After rolling, the magnesium alloy sheet was annealed, flattened, cut to size and then ground and polished on the same type of equipment used to surface copper or zinc plates. A portion of the batch was buffed and the remainder given a satin finish. To prevent tarnish during storage, the finished plates were oiled before wrapping in tissue and Kraft paper in the usual manner.

While the work to produce sheet of satisfactory quality was in progress, tests on the etching characteristics of various alloys in sheet form were made. An alloy that would deep-etch with a clean, smooth bottom was sought. The best results were obtained with an alloy containing 3 per cent aluminum, 1 per cent zinc, balance magnesium. Incidentally, this is the alloy composition used in Germany. It is probable that its good etching characteristics were a factor in the decision to change to this composition from the magnesium alloy containing 3 per cent zinc which was formerly used. The better corrosion resistance of

*Plate II—A halftone print direct from a magnesium plate.*

the alloy with 3 per cent aluminum, and 1 per cent zinc was also a factor in its selection.

## Preparation of the Plate for Etching

Photoengraving shops usually purchase their plate stock in standard sizes (of which there are many), the most popular being 0.065 by 22 by 28 in., 0.065 by 17 by 34 in. and 0.065 by 18 by 36 in. This stock is cut to the size required on guillotine shears and the polished or "good" side of the plate is scrubbed with a paste of pumice and water. This scrubbing is necessary to remove all traces of oil or grease that may be on the plate and also to provide a surface to which the light-sensitive enamel or "top" will adhere. It is an extremely important operation when working with zinc or copper and, with magnesium plates, the care taken during this step determines the success or failure of all subsequent steps in the process. If the scrub-up is not properly done, the enamel will not adhere and will lift off during etching. Improper procedure when scrubbing magnesium plates may also result in the formation of bubbles in "cold top" coatings which, in turn, will produce serious blemishes in the etched plate.

Although zinc and copper plates can be satisfactorily surfaced by scrubbing with a paste of pumice and water, it was found that magnesium plates must be scrubbed with a paste of pumice moistened with a 10 per cent solution of ammonium bichromate in water. The ammonium bichromate inhibits the reaction between the freshly cut magnesium alloy surface and the comparatively impure tap water used in most localities. The use of ammonium bichromate is especially necessary when the water supply is high in chlorides.

After thoroughly scrubbing the surface of the magnesium plate with the bichromated pumice, the pumice is removed by flowing and brushing the plate with a 10 per cent ammonium bichromate solution. After the excess bichromate solution is drained away and the plate very briefly rinsed, it is ready to be coated with the light-sensitive enamel or "top" which is preferably applied while the surface of the plate is still wet. Good results have also been obtained by applying the enamel to dry plates.

## Light-Sensitive Enamels or "Tops"

Tests showed that reproductions of line-cuts or half-tones on magnesium plates may be accomplished by employing glue, shellac or albumin tops. The glue, or "hot top," and the shellac, or "cold top," enamels seem to be the most widely used in this country and most of the experimental work on magnesium alloy etching plates was conducted with these two types of tops.

Hot top is a water emulsion of a good grade of fish glue containing ammonium bichromate. Cold top is a water emulsion of high grade shellac containing ammonium bichromate. There are many formulae for making cold top and many of them are unsuitable for use on magnesium because of reaction with the magnesium during the heating operation that dries the top on the plate. Cold top enamels that are strongly ammoniacal appear to be especially reactive.

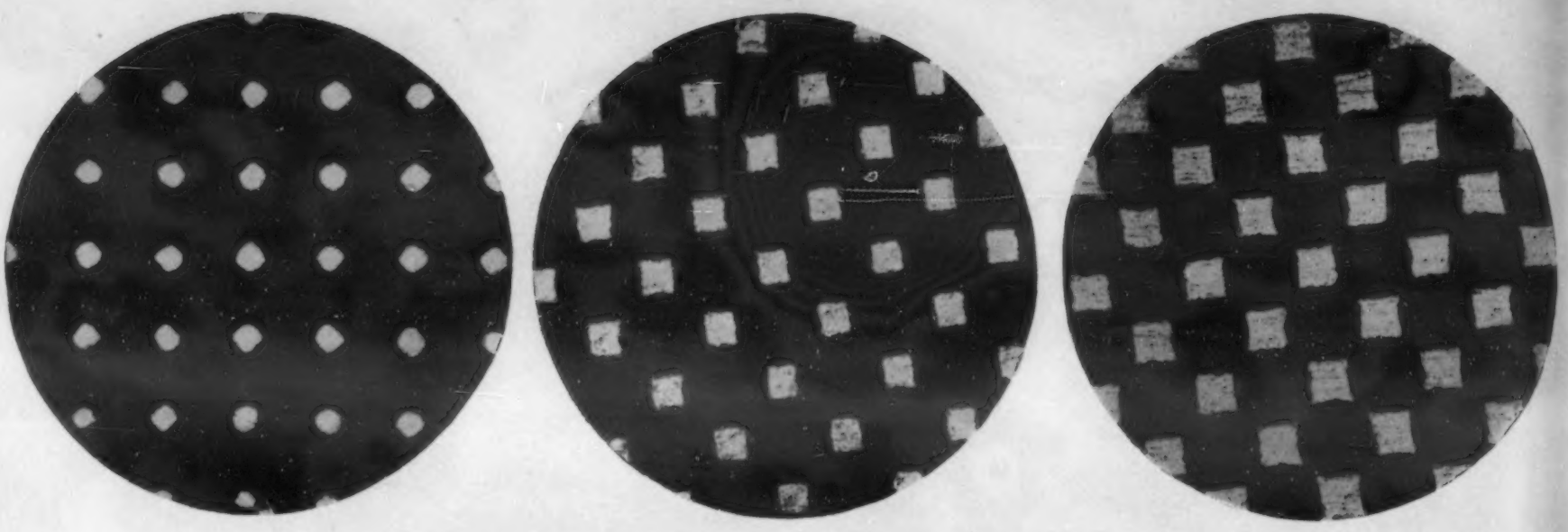
Both hot top and cold top have the consistency of thin varnish and are applied to the plate in the same way, i.e., by holding the plate at an angle and flowing the top over the plate from one corner until the entire surface is covered with an even film free from bubbles and specks of dirt. The film of top is finally dried by whirling it in a horizontal position over a gas flame. The temperature attained by the plate during the whirling operation seldom exceeds 140 deg. F. In most instances, plates are printed and developed immediately after coating but, if so desired, magnesium plates may be coated and stored as long as zinc or copper plates in a dark room. The so-called "dark reaction" is not accelerated by magnesium.

Plates coated by either hot top or cold top are printed in the regular manner by exposing them through a negative to ultra-violet light. Printing times of 5 to 10 mins. have been successfully used for magnesium plates. There is some indication that a slightly longer printing time is desirable for magnesium plates as compared to copper plates, but a skilled operator can soon determine the proper printing time for his particular machine after making a few plates.

The action of ultra-violet light produces a glue-bichromate complex that is insoluble in water or a shellac-bichromate complex that is insoluble in alcohol. Consequently, hot top plates are developed by placing them under a stream of water and cold top plates are developed by immersing them in a tank of alcohol and dye maintained at 70 deg. F. for a short period of time (usually about 2 mins.) and then placing them under a stream of water. It is customary to gently swab the plates with cotton while the water is flowing over them to aid development.

It was found that magnesium plates coated with hot top may be developed in the same manner as copper or zinc plates without any special precautions but magnesium plates coated with cold top may be ruined by leaving them in the alcohol too long or in the water too long. Either will cause the top to swell and lift off.

After developing and drying, the cold top plates are ready for etching but hot top plates must be "burned in," a process which involves heating the plates evenly and carefully (usually over a multi-burner gas stove) until the straw colored top turns brownish black. When working with magnesium,



*Figs. 1, 2 and 3. Plan view of original etched surface of zinc (Fig. 1), copper (Fig. 2), and magnesium (Fig. 3). 27X.*

it is usually desirable to harden the hot top prior to burning in. This step is frequently omitted when processing copper plates that are to be etched electrolytically in ferric chloride but nitric acid, the etchant used for magnesium, tends to "rot" some hot top enamels and, consequently, hardening is advisable to prevent this. Hardening baths containing ammonium bichromate, chromium alum, alcohol and water were found to be satisfactory.

During hardening, the bath is kept continually in motion in order to avoid small bubbles adhering to the plate. When a gray precipitate has formed on the uncoated portions of the plate, the glue is sufficiently hardened. Before burning-in, the hardened plate should be thoroughly rinsed with water and dried.

Magnesium plates withstand the burning-in operation without distortion and, because of the high melting point of the alloy (1160°F.) compared to that of zinc (786°F.), it will withstand abuse during burning-in better than zinc.

## Etching

Etching is probably the most important step in the process of reproduction by photoengraving and calls for a high degree of skill and artistic sense. This is especially true for halftones and color work where the shading, tone values and color of the finished print must match those of the original drawing, painting or photograph. This bit of legerdemain is accomplished by the etcher whose only tools are acid, a burnisher, a magnifying glass and a vivid imagination. Although depth of etch is important, the shade or tone is governed by the size of the dot. In high lights, the dot is etched to a pin point while in black areas, it is retained in practically its original dimensions, which, of course, depend upon the size of screen used to break up the stripping film negative photographed from the original copy. Various shades of gray or other color are obtained by the etcher adjusting the size of the dots according to his fancy.

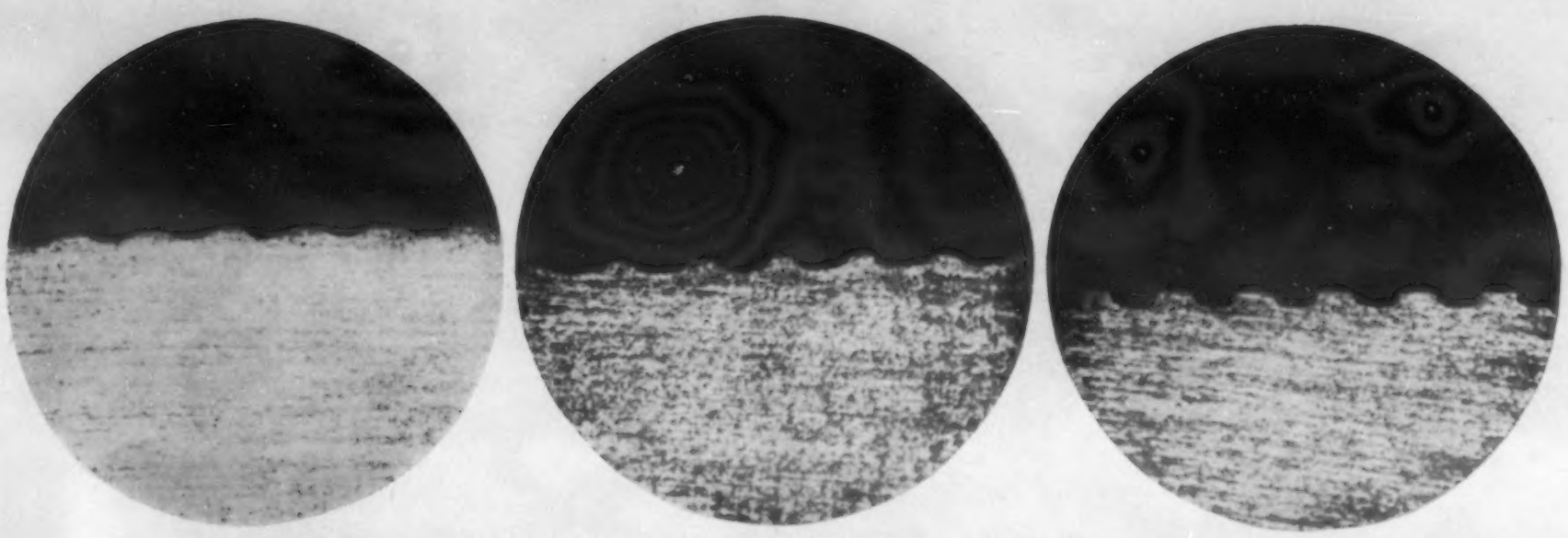
In this country it is almost universal practice to use machines for etching zinc. These same machines are suitable for the initial and sometimes for the final etching of magnesium plates. Re-etching by hand is necessary for high grade fine screen or color work, as with zinc or copper, but it can be accomplished more rapidly.

A few tests showed, however, that the acid concentration used to etch zinc reacted too vigorously with magnesium. Whereas the etching solution for zinc is made up of 1 part 40 deg. Bé nitric acid to 4 or 5 parts water, the most suitable etching solution for magnesium consists of 1 part 40 deg. Bé nitric acid to 15 or 20 parts water. Even with this dilute acid, etching proceeds very rapidly and an etcher usually over-etches the first few plates attempted.

The various steps in etching magnesium such as powdering up with a fusible resin called "dragon's blood" between "bites" are the same as for zinc. It may be desirable in some instances, however, to protect high light dots with an asphalt resist after the first etching or "bite." When the copy is a combination half-tone and line-cut and deep etching is necessary, the half-tone portion of the plate should be protected with asphalt. In other cases, it may be advisable to reinforce the whole top with an ink resist after the first bite.

For a given subject, the time required in the etching machine is about half that required by a zinc plate. With reference to copper, the time saved is, of course, much greater.

The same ratio of time saved also holds for the



*Figs. 4, 5 and 6. Cross sections of original etched surfaces of corresponding metals in Figs. 1 to 3. 27X.*

re-etching or hand etching step for high grade work. Re-etching is usually done with a small camel's hair brush and blotting paper. The acid used in the machine is too strong for re-etching. For this type of etching on fine screen half-tones, a solution containing 1 part 40 deg. Bé nitric acid to 39 parts water by volume has been used satisfactorily. If the rate of reaction is still too rapid, it may be reduced by adding a saturated solution of gum Arabic until the desired rate is obtained.

The etching characteristics of the plate material used in the photoengraving process has an important bearing on the degree to which faithful reproduction of copy may be attained. Since, in half-tone work, the dot is the basic structure of the reproduction, the type of dot formed, its uniformity and the degree to which it retains its original geometric pattern while being reduced in size determines the quality of the reproduction as a whole. At the present time, copper is preferred by the trade for high grade work where good detail and fine shading is desired. The reason for the better results obtained with copper as well as the reason why reproductions made with magnesium plates are at least equal to those made with copper plates is illustrated in Figs. 1 to 6 inclusive.[2] These figures are photomicrographs at about 27 diameters of identical spots on unused plates of zinc, copper and magnesium. Figs. 1 to 3 inclusive are plan views while Figs. 4 to 6 inclusive are corresponding cross-sections. The plates from which these photographs were taken had all been etched according to standard procedure for each of the metals but no re-etching had been done.

The deformation of the individual dots in the zinc plate (Fig. 1) is most conspicuous. Instead of having sharply defined outlines, their form is irregular, which will naturally affect the quality of printing. This is further illustrated by the cross-

*Figs. 7, 8 and 9. Cross section of etched surface of same metals after 54,000 prints. 27X.*

*Plate I—A halftone print direct from a magnesium plate.*

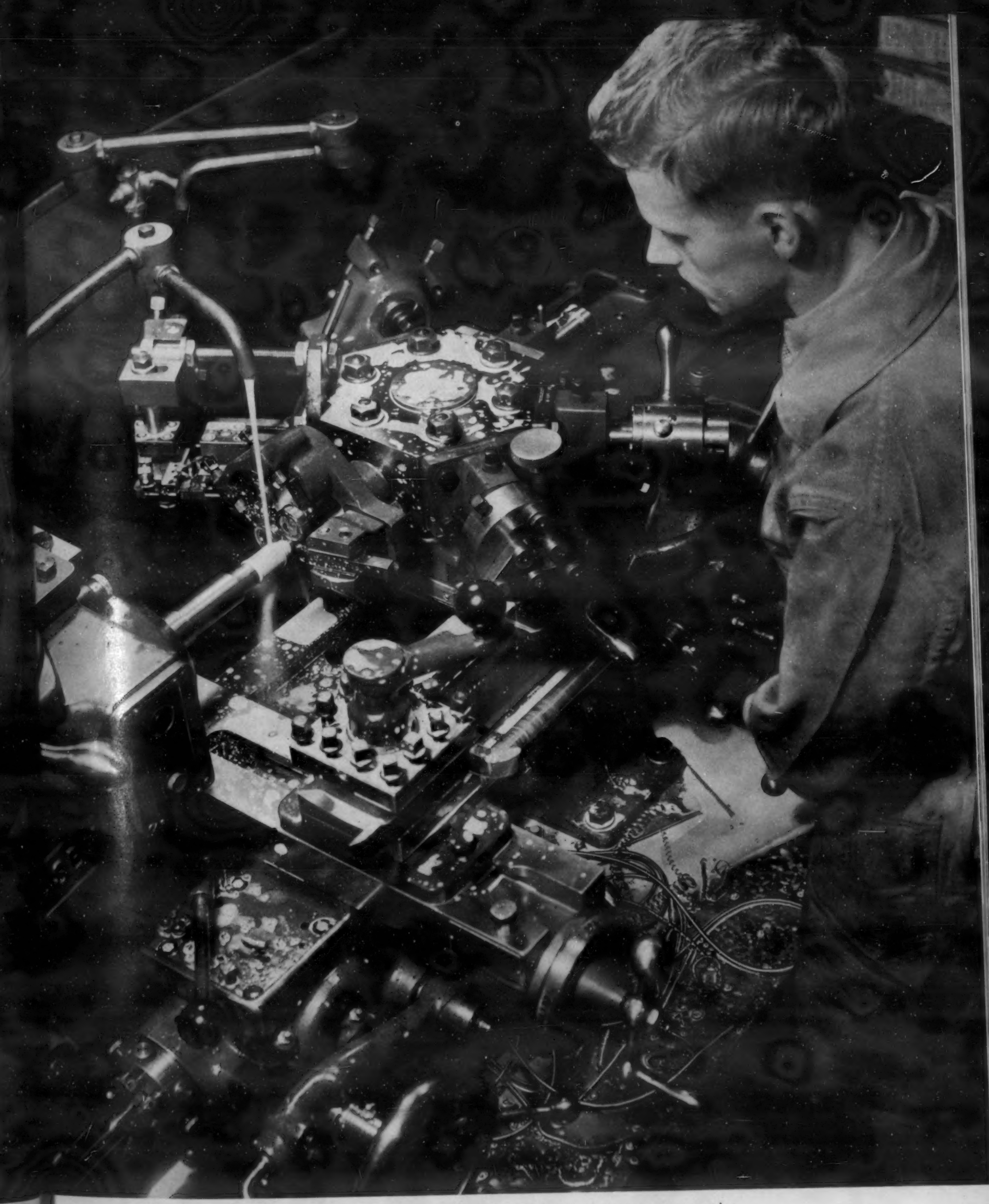


*Plate III—The same subject as Plate I printed direct from a copper plate.*

section view in Fig. 4, which clearly shows the surface of the dots to be rounded off, eroded or sloping thus causing the irregular pattern observed in the plan view.

The copper plate (Fig. 2) has dots which are considerably more uniform than those of the zinc plate and the tone values are, therefore, reproduced with greater fidelity. The cross-sectional view (Fig. 5) shows, however, insufficient depth of etching, calling for subsequent deep-etching. This operation requires additional time and may damage the high lights unless the work is done with great care by an experienced operator.

The dots in the magnesium plate (Fig. 3) show contours equally as sharp as those in the copper plate and the gradation of tone values is as completely retained as in the copper plate. The cross-section view of the magnesium plate (Fig. 6) shows the uniformly good depth of the depressions. Such depth of etching cannot be produced in copper or zinc without subsequently deep-etching.

It must not be assumed that copper and zinc plates with sufficient depth of etch are not regularly produced. The foregoing merely brings out the fact that sufficient depth of etch is more quickly and more easily obtained in magnesium plates.

Unless repairs are necessary, the etching operation completes the photoengraving process and the plates are ready to be mounted on wood blocks for printing after beveling and routing, if necessary. If the plates are not used for direct printing electrotypes or mattes are made. The result of all this painstaking work is shown in Plates I and II which are 133 line halftones printed direct from magnesium plates. Plate III is the same subject as Plate I printed from a copper plate. The magnesium reproduction is seen to be at least equal to the copper reproduction.

## Repairing Damaged Plates

Completely etched plates frequently must be repaired or touched up with graving tools before a satisfactory proof is obtained. The plate may be scratched from careless handling or a small defect in the top may have resulted in one or more missing dots. In color work, the match may not be perfect and portions of one or more of the plates may have to be cut out and soldered into correct position. Long experience and great skill have perfected methods of making such repairs on copper or zinc plates.

Although the same methods used to bevel and rout copper and zinc may be used equally well on magnesium plates, repairing and soldering require an entirely different technique. For instance, a small scratch or a few missing dots in a copper plate can be easily repaired by raising small burrs with a sharp pointed tool in the locations dots should be. These burrs are then burnished level with the general surface of the plate and the dots shaped to proper size by a graving tool. This operation is quite difficult with magnesium because the burrs tend to break off if a row of closely spaced dots must be raised. Isolated dots may be raised with a tool and rubbed down to correct level with sufficient skill but large defects are best repaired by soldering followed by graving the soldered area to correct tone value.

The soldering of magnesium plates requires a different technique than that used for copper or zinc but effective soldering is not especially difficult. A special solder is used and the magnesium surfaces to be soldered are "tinned" before complete joining is effected. One satisfactory solder contains 72 per cent tin and 28 per cent cadmium (AMC Solder A). No flux should be used. To "tin" the magnesium surfaces successfully, they must be clean and bright and the temperature of the magnesium must be above the melting point of the solder. A small wire brush used to abrade the surface of the magnesium under the solder expedites the "tinning" operation.

Complete information on repairing all types of defects is too lengthy to be given here, but a recent pamphlet by F. Lührig on the "Processing of Photoengravings on Elektron Etching Plate 28"[3] described experiments with magnesium plates and concluded: "Every mechanical after-treatment can be carried out with magnesium plates without difficulty. The soldering of magnesium can be carried out well by following directions and after acquiring some practice."

## Service Life of Magnesium Plates

Magnesium alloy etching plates have a hardness greater than that of zinc and approximately the same as that of copper plates, but tests have shown that, when used for direct printing magnesium plates stand up better than copper plates. This is illustrated by Figs. 7 to 9 inclusive[2] which are photomicrographs at about 27 diameters of identical areas in similar zinc, copper and magnesium plates taken after making 54,000 prints under the same conditions on the same press. The better condition of the magnesium plate is obvious. As many as 90,000 prints have been made with magnesium plates without causing enough wear to impair the quality of the reproduction.

Because of the fact that magnesium plates etch without under-cutting the top, they produce excellent mattes in either paper or lead and a great many impressions can be made before distortion occurs.

## Discussion and Conclusions

When the work on magnesium photoengravers' plates was started about 3 yrs. ago, it was known that they were being used on an increasingly large scale

in Germany. It was not certain, however, whether the growing use was due to the advantages of magnesium or to the scarcity of copper and zinc in Germany. It is our present opinion that magnesium plates offer a new and perhaps better medium of reproduction and they should be of considerable interest to the progressive photoengraver in this country. On the other hand, it is realized that general acceptance of the magnesium plate may be rather slow because of the different shop practice recommended for magnesium plates and the extra care that must be taken in handling magnesium plates from the time the sheet is received from the manufacturer to the time when the finished plate is dispatched to the printer.

So far as shop practice is concerned, the most serious obstacle to the general acceptance of magnesium plates by photoengravers is the different and more difficult technique required when repairing damaged plates. It has been pointed out by photoengravers that the time saved in etching magnesium plates may be offset by the extra time required to clean up or repair slight defects. The development of simple and rapid methods of repairing magnesium plates would be of great aid in the commercial introduction of magnesium etching plates and present experimental work is concerned with this problem.

The tendency of magnesium to tarnish or pit under unfavorable conditions of storage has caused some concern for plates that are used and then filed away for possible future use. Although this is a problem to consider, it is felt that it can be solved without particular difficulty. Under ordinary indoor conditions, it is known that 3 yrs.' storage does not adversely affect etched magnesium plates. Where acid fumes or excessive humidity are present, the plates can be protected by a chemical dip coating or by oiling. Corrosive attack on magnesium requires the presence of moisture so the essential point in storing magnesium etching plates is to keep them in a dry place and to make sure that they are dry before storage.

From our experience thus far, a partial list of the advantages or benefits that would result from the use of magnesium etching plates are:

1. Magnesium plates can be processed by all three commonly employed light-sensitive tops.
2. Magnesium plates are suitable for line-cuts, coarse screen or fine screen work and thus capable of replacing both copper and zinc, allowing the plant to standardize on one plate material and one processing method.
3. Magnesium plates can be processed more rapidly than either copper or zinc plates.
4. Magnesium plates allow longer runs when used for direct printing.
5. Magnesium plates will make more and better mattes.
6. Magnesium plates weigh less than 1/4 as much as zinc plates and less than 1/5 as much as copper plates which would result in greatly lowered mailing cost to firms that ship plates long distances.

Although it is felt that a little more experimental work is desirable before offering magnesium plates to the trade, the economic side of the picture has been given some consideration. Calculations indicate that on an area basis, the selling price of magnesium plates would be higher than that of zinc plates and slightly lower than the current price for copper plates. Although the metal in a finished etching plate represents a very small proportion of the total cost, it is a factor that must be considered and helps to determine the type of work on which magnesium is most likely to be used. Indications at present are that magnesium might replace zinc in plants where utmost speed in processing is desirable and where the higher quality of reproduction is considered worth the extra cost. It might replace copper for high grade work in plants where greater speed and lower cost of processing are desired with no sacrifice of quality.

In conclusion, it is hoped that this article will stimulate an interest among photoengravers in magnesium etching plates which appear to the layman to have many desirable characteristics. Although magnesium plates are not being marketed to the trade in general, the American Magnesium Corp. will be pleased to cooperate in tests by interested engravers under controlled conditions.

## Acknowledgments

The author wishes to acknowledge the very considerable aid in this work rendered by Roy Manny of the Rolled Plate Metal Co., Charles Hoyer of Fuller & Smith & Ross and Messrs. Friedey and Pogalies of the Ad Service Engraving Co. The magnesium sheet was ground and polished by the Rolled Plate Metal Co. of Brooklyn, N. Y. The magnesium cuts to illustrate this article were prepared by the Ad Service Engraving Co. of Cleveland. The encouragement and suggestions made by Mr. Hoyer during the course of the work were most helpful.

### *References*

[1] B. W. Gonser and S. Epstein. "Metals and Alloys in the Printing Industry." *Metals and Alloys*, Vol. 8, Jan., Feb., Mar. and Apr., 1937.

[2] Courtesy I. G. Farbenindustrie, Bitterfeld, Germany.

[3] Die Herstellung von Klischees auf "Elektron Aetzplatte 28," by F. Lührig. *Mitteilung des Forschunginstituts für das graphische Gewerbe.* Technische Hochschule, Berlin.

# Pinhole Corrosion in G

by W. M. PEIRCE and G. C. BARTELLS

*Chief, Research Division, The New Jersey Zinc Co., Palmerton, Pa., and Field Engineer, American Zinc Institute, Collinsville, Ill., respectively.*

*Pinhole corrosion in galvanized watering tanks has been on the increase in recent years. A study of the cause of this was instituted by the American Zinc Institute. The problem was attacked in cooperation with the research department of The New Jersey Zinc Co.*

*This article gives the results of the study of this problem. Tin was found as the source of the trouble, at least when it is present above some safe limit lying below 0.50 per cent.—The Editors.*

THE NORMAL TYPE OF FAILURE of galvanized steel is a very uniform corrosion of the coating and later of the base. Variations in thickness of coating are reflected in earlier disappearance of the coating and earlier failure of the base where the coating is thin but these variations are gradual over relatively large areas. Pitting is not normal. The electrolytic protective effect of zinc on iron opposes a pitting type of corrosion.

The occurrence, more common in recent years, of pinhole corrosion in galvanized watering tanks, used for watering stock, has therefore been a puzzling problem. Several years ago the American Zinc Institute assigned one of the authors to investigate the causes of the erratic behavior of galvanized steel in this type of service.

## Differences in Water

The most obvious possible source of variation in type of corrosion and useful life appeared to be differences in the water. This explanation was quickly ruled out by a number of observations of tanks used with a single water supply which exhibited extreme differences in behavior.

The possibility that pin-holing was due, where it occurred, to fortuitous accumulations of foreign matter was also explored. Here again no correlation could be found between the presence of either inorganic materials or organic slime accretions and abnormal corrosion. There was evidence, however, as would be expected, that such foreign materials may serve as the focal points for pitting in tanks susceptible for another reason to a pitting type of corrosion.

*Galvanized material after 32 yrs. of service. Tin content, 0.02 per cent.*

Many other factors were considered such as the flow of water in the various tanks examined which might affect the degree of aeration or give rise to comparatively stagnant areas. All such speculations proved to have no supporting evidence.

## Fourteen Samples Examined

During these investigations samples had been secured from a number of good and bad tanks (the former with some difficulty since it involved cutting into tanks still in service). Ultimately seven samples

# in Galvanized Watering Tanks

*Galvanized material after 1½ yrs. of service. Tin content, 0.69 per cent.*

were secured from tanks which had given satisfactory service for an average of 22.7 yrs. (one over 32 yrs.) and seven samples from tanks which had pitted through in an average of 1.4 yrs. (maximum life 3 yrs.) These samples were turned over to the Research Division of The New Jersey Zinc Co., for examination.

Visual examination failed to give any clue to the possible cause of pitting on the bad samples. The coatings were then stripped and analyzed. The stripping of the inside and outside surfaces of each tank sample was done separately in order to compare the relative loss of coating under atmospheric exposure versus total immersion in each case.

## Tin Content and Pitting

The results of the coating analyses showed a startling correlation between pitting and tin content of the coating. Table 1 shows the results of the analysis and weight of coating determinations. It will be noted that not one of the good tanks analyzed more than 0.28 per cent and not one of the bad tanks less than 0.54 per cent tin. A perfect correlation, as exists here, with this number of samples representing as many different conditions of service is strong evidence that the tin content is in fact the primary factor leading to pinhole corrosion of galvanized sheets under conditions of total immersion.

There are, it happens, sound theoretical grounds on which to base an explanation for this behavior. Tin is present in a tin-zinc alloy as free tin in the grain boundaries. Electrolytically, tin is known to accelerate the corrosion of both zinc and iron and may be expected to behave similarly with respect to the alloy layers whose potentials are between those of zinc and iron.

*Table. 1—Results of Analysis and Weight of Coating Determinations*

| Sample No. | Condition | Service, Yrs. | Wt. of Coating Outside of Side Wall | Per Cent | | | |
|---|---|---|---|---|---|---|---|
| | | | | Pb | Cd | Al | Sn |
| 102 | Badly pitted | 1½ | N * | 0.60 | 0.43 | 0.010 | 0.69 |
| 104 | Badly pitted | 1½ | 0.54 | 0.60 | 0.33 | 0.0025 | 0.77 |
| 107 | Pitted | 1 | 0.52 | 0.56 | 0.31 | 0.0027 | 0.68 |
| 112 | Pitted | 1 | 0.58 | 0.39 | 0.42 | 0.0020 | 0.57 |
| 106 | Pitted, partly rusted | 3 | 0.55 | 0.61 | 0.32 | 0.0040 | 0.54 |
| 111 | Pitted, partly rusted | 1 | 0.52 | 0.41 | 0.52 | 0.0032 | 1.0 |
| 113 | Pitted, partly rusted | 1 | 0.47 | 0.52 | 0.11 | 0.0033 | 1.20 |
| 103 | Partly evenly rusted | 25 | 0.76 | 0.68 | 0.19 | 0.0037 | 0.12 |
| 110 | Inside evenly rusted | 14 | 0.47 | 0.74 | 0.36 | 0.0032 | 0.14 |
| 105 | Some yellow rust | 25 | 0.62 | 0.66 | 0.27 | 0.0033 | 0.28 |
| 101 | Good | 27 | 0.37 | 0.55 | 0.31 | 0.0035 | 0.11 |
| 109 | Good | 13 | 0.57 | 0.50 | 0.30 | 0.0047 | 0.05 |
| 108 | Good | 23 | 0.60 | 0.54 | 0.31 | 0.0043 | 0.14 |
| 114 | Good | 32 | 0.52 | 0.61 | 0.09 | 0.0020 | 0.02 |

* N = No sample.

*Table 2.—Relationship Between Tin Content of Coatings and the Rate of Corrosion*

| | Average Weight of Residual Coating on (1) Good Samples (.02—.28% Sn) | (2) Pitted Samples (.54—1.2% Sn) |
|---|---|---|
| (1) Both sides: Bottom and side wall ..... | 0.48 oz. per sq. ft.* | 0.36 oz. per sq. ft.* |
| (2) Outside—side wall | 0.56 oz. per sq. ft.* | 0.53 oz. per sq. ft.* |
| (3) Inside—side wall. | 0.38 oz. per sq. ft.* | 0.19 oz. per sq. ft.* |

* Oz. per sq. ft. of surface.

Average Years of Service:
Good samples —22.7
Pitted samples— 1.4

Apparent Increase in Zinc Loss from Inside over Outside of Side Wall Per Year of Service:
Good samples—0.008 oz. per sq. ft. of surface
Pitted samples—0.24 oz. per sq. ft. of surface

Electrolytic effects are normally more pronounced under conditions of total immersion where this abnormally rapid corrosion has been observed. This theory is also consistent with the pitting type of attack. It should be noted, however, that the general rate of corrosion of the coatings has been accelerated more by total immersion in the case of the high tin coatings. See Table 2.

## Use of Tin in Galvanizing Has Expanded

Another related fact is of interest. The use of tin as a means of controlling spangle has increased sharply since the war and this type of corrosion trouble has also attracted more attention. The statistics on the use of tin in the galvanizing industry indicate an average use of nearly 0.50 per cent on all products in 1938 which, of course, means a higher average on sheets while in 1928 the average was only 0.05 per cent.

The evidence at hand points strongly to tin as a source of trouble, at least when present above some safe limit lying below 0.50 per cent. The evidence is confined to service under total immersion conditions but the effect may be present to a less extent under other conditions of service.

Further tests are contemplated and it is probable that some existing data on service and exposure tests can be profitably reexamined for a possible correlation with tin content where samples are still available.

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*Note:* Since this paper was originally prepared, 13 additional samples have been received and the coatings analyzed. Seven of these having an average service life of 27 years analyzed between 0.11 and 0.27 per cent tin. Six having an average service life of 1.3 years, analyzed between 0.31 and 1.4 per cent tin. These results furnish ample confirmation of the data and conclusions given in the article.

# Stainless Steel in a Hollywood Film Processing Laboratory

## A Pictorial Presentation

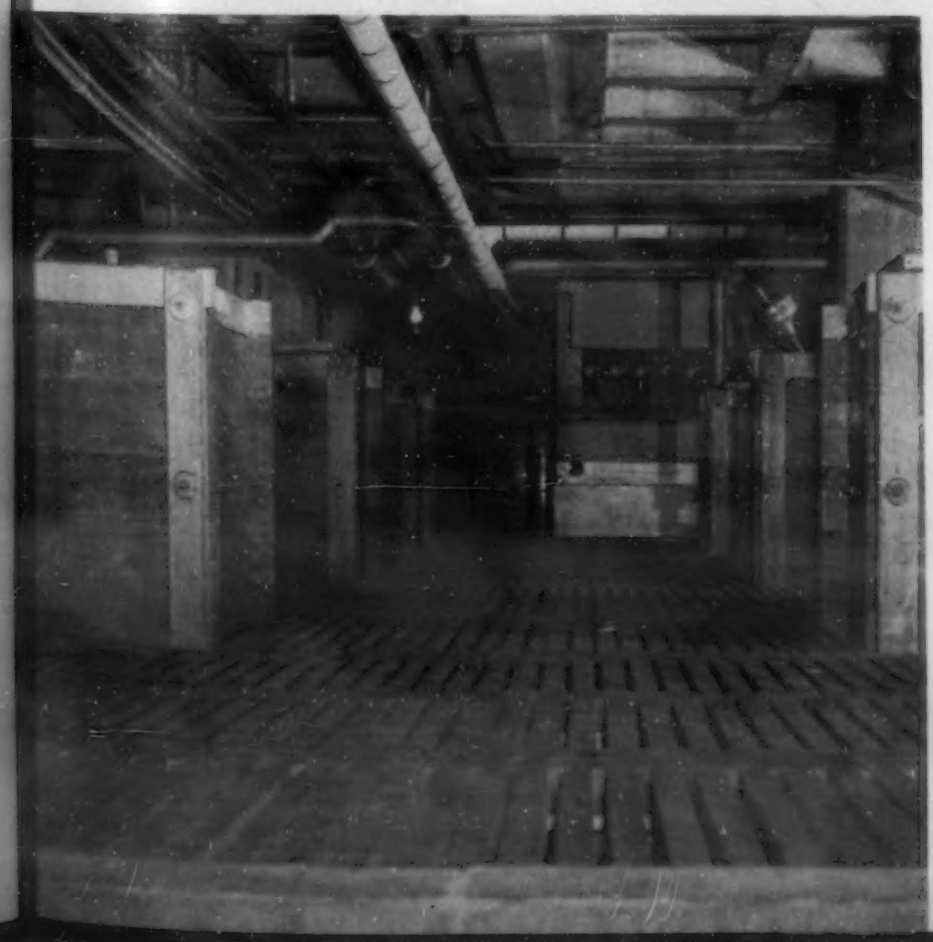
A FILM PROCESSING LABORATORY for Warner Bros.-First National in Hollywood, Calif., has recently been completed at a cost of $500,000. It is said to represent the "last word" not only in photographic finishing equipment, but in the broad scale application of stainless steel to the exacting conditions to be encountered in handling the corrosive developing and fixing solutions necessary in photographic work.

As shown in the accompanying sequence of pictures, the stainless steel has been used for developer mixing vats, tubes, valves, and pumps; for heat exchangers and filters of the pumping system itself; for the cylinders, reels and spools in the darkroom; and for the machines used in the film cutting department. Wherever chemicals come in contact with the metal, "Enduro 18-8 SMO," as made by the Republic Steel Corp., stainless, has been used. Elsewhere "Enduro 18-8" stainless has been installed.

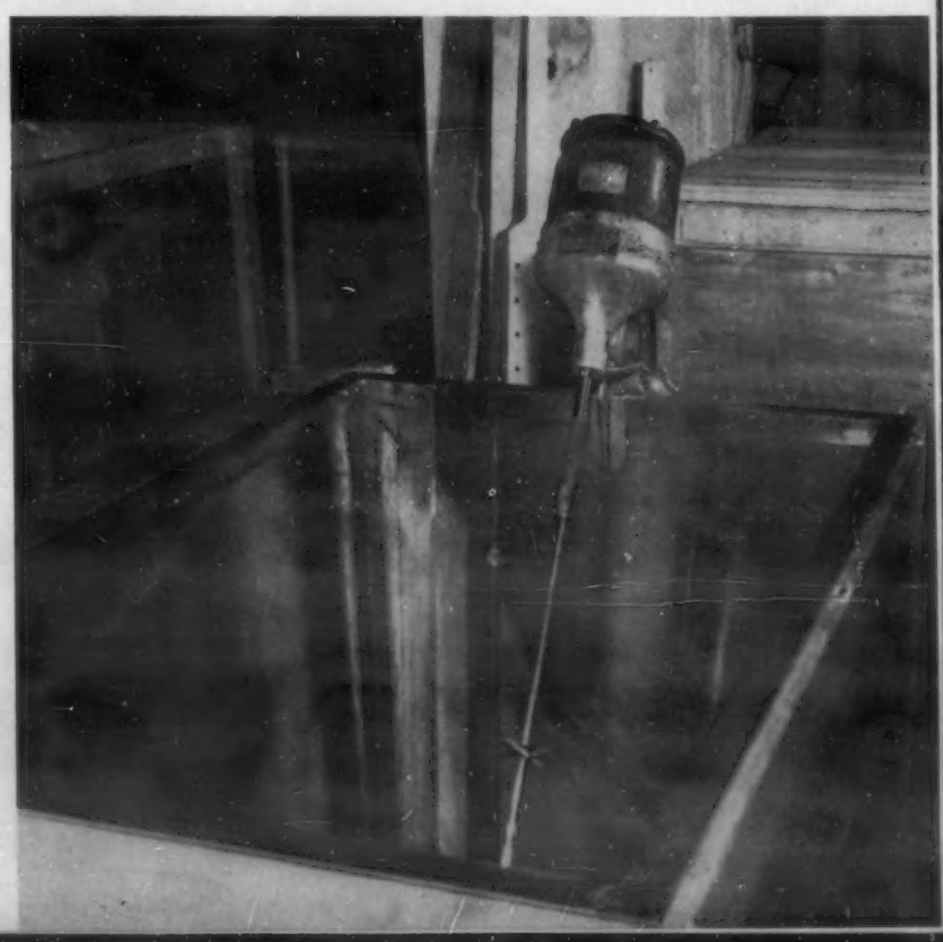
In general, regardless of the size of the photographic processing installation, whether it be on the mammoth Hollywood scale depicted here or on the tiny scale typified by the "home" darkroom of the candid camera enthusiast, one of the most severely corrosive conditions to be met is in the handling of the chemical solutions used in the various kinds of fixing baths. Here there is always an acid condition with sulphites, thiosulphates, alums, and, occasionally, chlorides present as the active corrosive constituents in water solution.

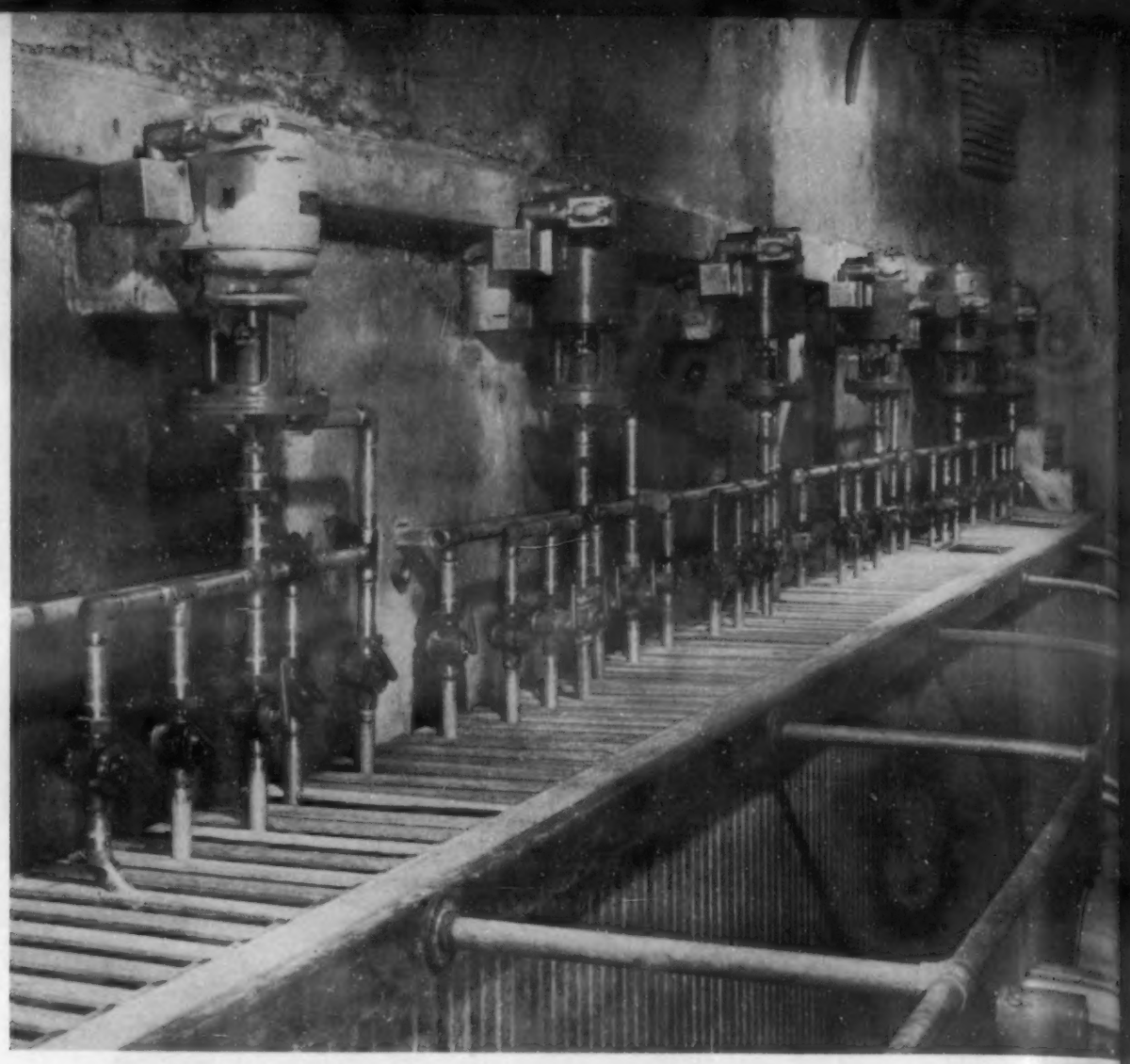
Developers may contain sulphites, sulphates, bromides, iodides, and borates as their more corrosive constituents. However, such developers contain organic compounds which, together with the basicity of the solution, render such solutions less corrosive than fixing baths.

*In the new film processing laboratory of Warner Bros.-First National in Hollywood, the developer mixers are made from wood and lined with Enduro stainless steel. The concentrates are mixed in these tanks prior to entering the pumping system.*



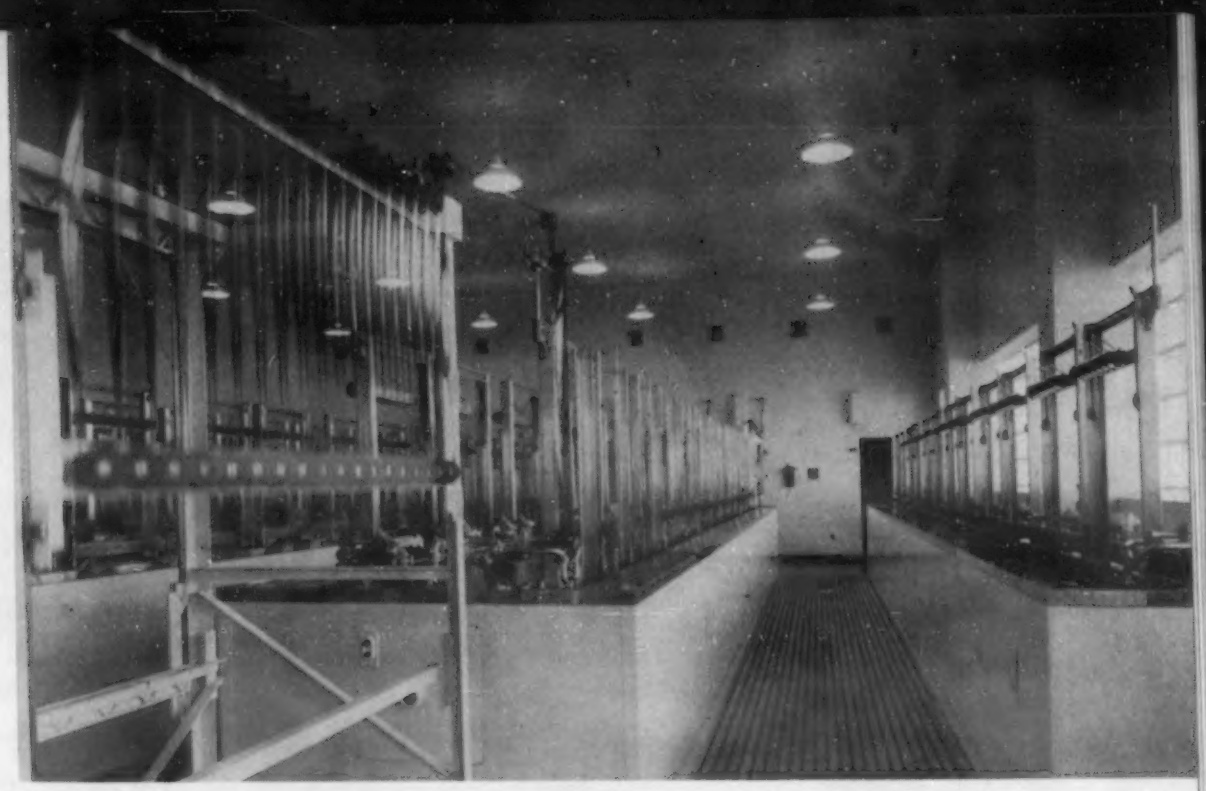
*Close-up view of the stainless steel lined concentrate vat showing motor driven stainless steel shaft used in mixing the powerful developing solutions in the new $500,000 film processing laboratory of Warner Bros.-First National.*



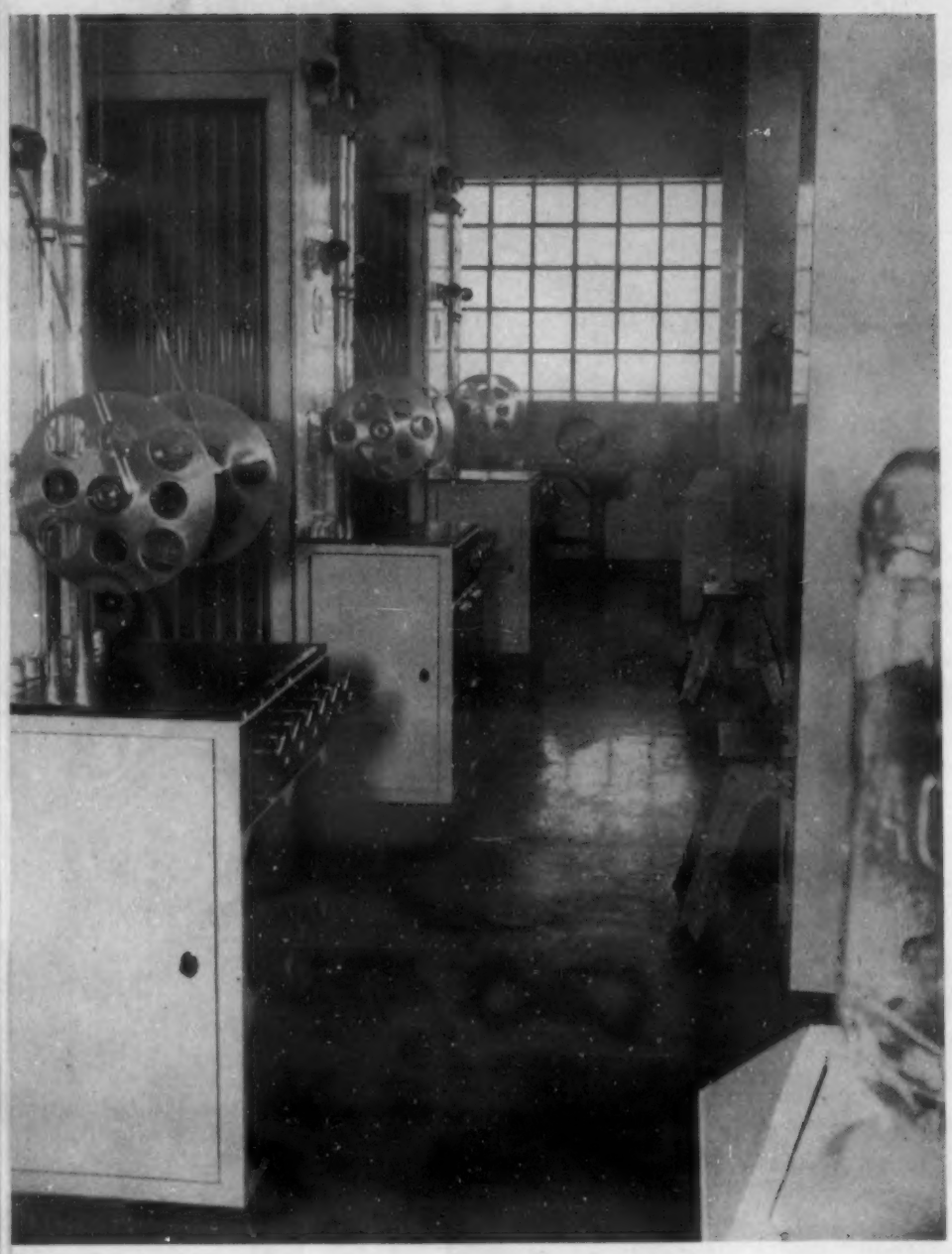


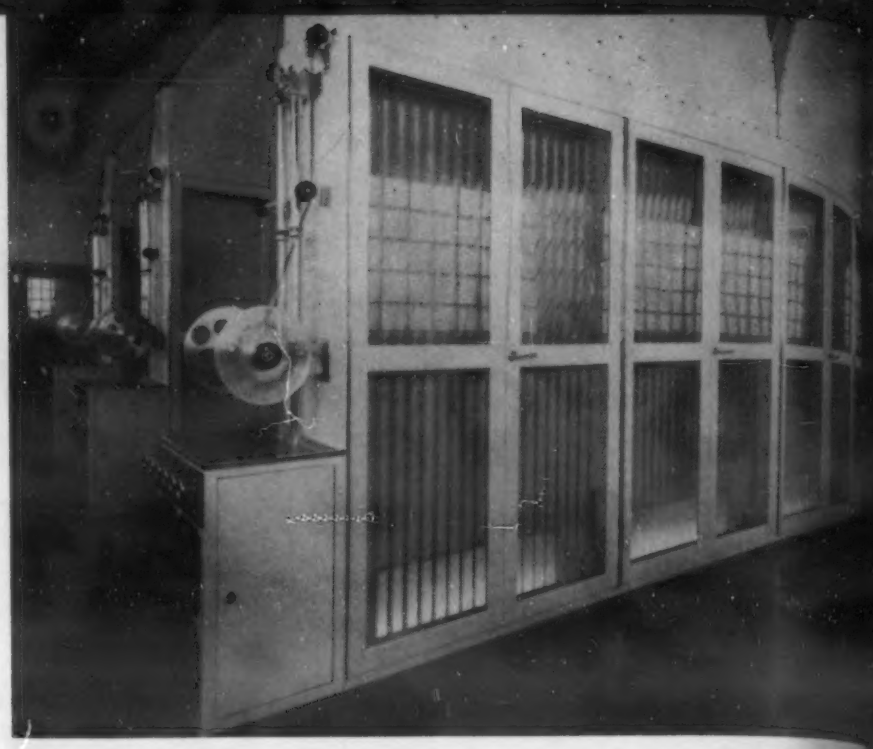
*Tubes, valves, pumps, heat exchangers and filters are all fabricated from stainless steel in the new film processing laboratory of Warner Bros.-First National.*

*Taken by flashlight exposure since this film developing room is of course always dark, the installation of cylinders, reels and spools all made of stainless steel can be seen on both right and left elevators. Film is run continuously through the developing and hypo tanks which are also lined with Enduro. After one water rinse the film passes through apertures in the wall (shown in background) to the next process.*



*Three views of the washing and drying machines all of which are fashioned of stainless steel. The wash is plain water. Drying temperature is approximately 74 deg. F.*





*The finished film is coiled on take-off reels in the new film processing laboratory of Warner Bros.-First National, Hollywood.*

*Left: Close-up of the take-off reels on which finished film is coiled in the new film processing laboratory of Warner Bros.-First National, Hollywood.*

*Negative cutting is extremely important. As shown here the cutting equipment in the new Warner Bros.-First National film processing laboratory is made almost exclusively from stainless steel.*

*The technical "fade" of one scene into another in modern motion picture production is carefully and smoothly performed in the "Chemical fade" laboratory. Here as elsewhere in the new film processing laboratory of Warner Bros.-First National in Hollywood, stainless steel is used extensively in equipment with which film and chemicals must come in contract.*

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# Metallurgical Control in the Fourdrinier Wire Industry

by D. C. DILLEY

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ORIGINALLY A HIGHLY SKILLED ART, the manufacture of the Fourdrinier wire has in recent years been thoroughly revolutionized by scientific control. Wire for these fine meshes is one of the highest quality wires now being produced. Looms have developed from small, clumsy, hand driven affairs, to modern electric powered, automatic ones. The older joining methods whereby the woven wire mesh is joined to form the endless belt, the heart of the Fourdrinier machine, have developed from sewed seams, to soldered seams, and more recently to electric and gas welded seams. A partial bibliography of related subjects listed with this paper indicates only too well the small volume of literature available in this field. William R. Murphy[9] has outlined the sequence of manufacturing operations from raw material to finished product in a modern Fourdrinier manufacturing plant. Because the success or failure of a wire from the engineering point of view depends upon its reaction as a metal product to the destructive forces which act upon it, an attempt will be made to illustrate what precautions are taken to insure that each Fourdrinier wire leaves the producing plant with the best physical properties which to our knowledge will insure its maximum efficiency.

## The Raw Material

H. E. Brown[2] has given an excellent account of the problems in selecting materials for Fourdriniers. For years the standard material was 80-20, mixture low brass, which has recently been supplanted in many mills by bronze of the 8 tin, 0.3 per cent phosphorus variety, in the warps, and a lower tin alloy bronze in the shoots.

Probably no wire product is confronted with more varied destructive conditions of service than is the Fourdrinier wire. Fatigue by bending, under the action of corrosive paper stocks—combined with abrasion from suction box covers makes the list of suitable metallic materials a small one. Phosphor bronze and brass wire have maintained their undisputed position in the field. No field of metallurgical research shows more need for a new alloy than does the Fourdrinier wire industry. The day for the introduction of a new material is long overdue. When it does come, the material will probably combine the corrosion resistance of the stainless steels with the ductility and resistance to work hardening of the wrought phosphor bronzes.

In the discussion which followed a recent technical paper on "Stainless Steel Wire"[12] at the 1938 Wire Association meeting, the possibility of the use of this alloy steel for Fourdrinier wires was brought up. This seems logical enough for its resistance to corrosion is excellent and the warp knuckles should show little wear for the more they were abraded the harder they would become. It is this same property of rapid work-hardening however, which defeats the use of this material in the field.

Screen wire in stainless steel will always have its place in corrosion resistant applications, in locations where little flexing is required. At its present stage of development it is unlikely that it will become applicable to the Fourdrinier wire.

## Corrosion Fatigue

The whole problem centers around one particular type of wire failure known as "corrosion-fatigue." It is a known fact that the fatigue limit of high tensile steels is materially lowered when the reverse-bend life tests are made in such an atmosphere as a salt spray. Likewise, the fatigue limit of many materials is noticeably increased when such tests are made in vacuo. The effect of a corrosive atmosphere in these tests is not thoroughly understood, but it is believed that the formation of even sub-microscopic cracks[14] on the surface of the materials is possible. This notching effect correspondingly

reduces the cross-sectional area of the specimen causing stress concentrations, overloading, and premature failure.

In connection with this discussion of materials for the Fourdrinier wire, attention to the conclusions of H. J. Gough and D. G. Sopwith[13] in their article titled "The Resistance of some Special Bronzes to Fatigue and Corrosion Fatigue," should be called. Discussing the results of their tests the authors concluded that, while some of the alloys tested had higher endurance limits in air than phosphor bronze, the latter had a better corrosion fatigue resistance and that phosphor bronze was exceeded only by beryllium copper in this respect. The position of these beryllium alloys is now firmly established in many fields of engineering practice, but unfortunately, the price of the metal as well as certain fabrication difficulties prohibit their use in the Fourdrinier wire at the present time.

Phosphor bronze retains its position by right of laboratory investigation as well as mill life records. Until some new alloy is developed which will show higher corrosion fatigue than this alloy, the life of the Fourdrinier will very probably not be materially extended beyond its present range.

## Manufacture of Weaving Wire

Most manufacturers produce their own wire for weaving purposes. The great advantage secured from maintaining a wire shop at the plant can be understood when one realizes the variation in wire diameters which are required to meet the strength, mesh, drainage, and other requirements of the 900 or more paper machines in the country. As many as 200 sizes and types of wire must be on hand for weaving in a large shop. These vary, generally from 0.004 in. to 0.018 in. in diameter, the sizes changing in quarter-thousandths inch on the diameter. When new products appear on the paper market, new Fourdrinier must be made to produce these products, and the change in Fourdrinier wire specifications for the particular machine begins in the wire shop.

Wire is generally received in 14-in. coils at from No. 15 to No. 20 B & S gage annealed soft for redrawing. (Few shops produce their own alloys, or maintain a rod mill to supply heavy wire for the finish drawing shop). In current practice this material is in 150-lb. coils, either bright annealed or pickled following the older pot type anneal.

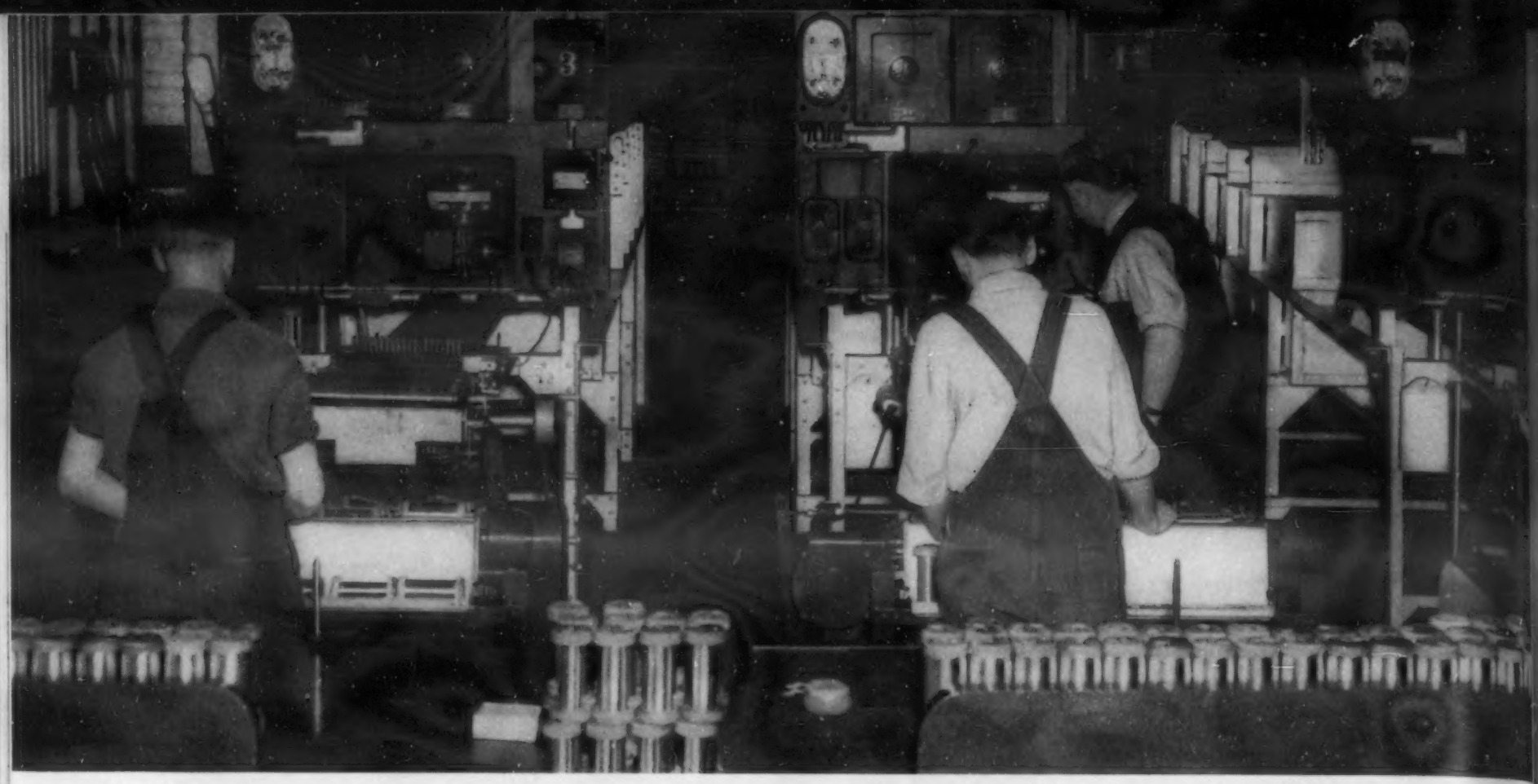
The original coils are analyzed chemically either in a large commercial laboratory or in a small chemical laboratory maintained at the plant. Those coils which in any way prove to be inferior in quality are returned to the original source. Cleanliness of the material is also checked by means of the metallurgical microscope, where any inclusions or faults in the structure of the wire will appear when the grain size of the metal is checked to ascertain the extent of the softening anneal. When once accepted the shipment starts its travel through the endless wire drawing equipment, each coil being welded to the one directly preceding it by an electrical welding unit, thus increasing the efficiency of drawing.

## Drawing Through Diamond Dies

Break down wire drawing machines (utilizing 8 to 10 diamond dies) take the raw material at approximately 0.060 in. dia. and reduce it to approximately the cold working limit of the material being drawn. The extent of original drawing affects the strength of the finished wire, so great care must be taken to insure uniformity of processing. Wire enters the machine and passes through a diamond die of the next smaller gage size, and then to the next capstan where it is looped around this roll several times to insure the required tension for drawing. At the take-up end of the first drawing machines the wire has been reduced to some size approximating 0.020 in. dia. It is coiled on 8-in. blocks at the take-up end, and taken in 40-lb., tied, coils to be softened by annealing before being reduced to sizes suitable for weaving.

Steam or gas atmosphere furnaces protect the surface of the wire from excess oxidation when it is being annealed. Continuous strand-type, moving hearth, and stationary pot furnaces are in use for the process annealing. The temperature of anneal (about 1000 deg. F.) is controlled automatically by suitable indicating-recording potentiometer controllers. The time of the anneal is important, for wire which might be suitable for other products, may be incorrectly annealed to be used for weaving wire.

Following this process anneal, a slight pickle in acid is often given to insure an absolutely clean surface for drawing to the finished sizes. The modern wire shop employs a man to constantly check on the condition of the drawing compound, and to insure maximum diamond die efficiency by repolishing the bore of these stones as soon as they show any slight imperfection. In this respect, it is interesting to note that the content of soap and fat in the lubricant, the rate at which the wire is reduced, and even the angle in the bore of the diamond die all have an effect upon the resulting physical properties of the weaving wire. The machine operators must constantly check the diameter of the finished wire, and must renew the finishing die as soon as it shows any indication of drawing "off size." Thus the control of all processing through daily measurements of these factors has brought even the older less-skilled operations of wire manufacture under laboratory control.



*Strand annealing of weaving wire. The panel board shows continuous indication of the furnace temperature. A secondary circuit of the thermocouples makes a continuous chart of all annealing operations, thus checking actual control instruments, and making a record of annealing temperatures.*

## Final Critical Annealing

Wire is finish drawn from the process annealed coils on to steel spools. This wire is then ready for the final step in processing. The material is in the hard drawn state, and it then receives its final critical annealing to make it soft enough for the required weaving operations, and yet hard enough to withstand the various loadings placed on the finished wire cloth in the paper machine. The anneal is accomplished in multiple strand furnaces, the wire being pulled through alloy tubes, surrounded by a suitable heating muffle. The finish remains bright and clean for an atmosphere in the annealing tubes excludes all oxygen, water vapor and sulphur compounds which might tarnish the product.

The wire is unreeled and passes continuously through the tubes at speeds varying from 80 to 500 ft. per min., and respooled at the other end of the furnace. In this operation, the elongation of the wire has been found to be a good indication of its physical properties. The furnace operator, therefore, takes a rough check on the elongation of every spool of wire annealed in the furnace. At this point a careful daily check must be made to insure a uniform quality of the weaving wire.

Representative samples of the wire shop daily production are checked by a laboratory staff for roundness, surface characteristics, etc. A tensile testing machine designed especially for fine wires is used to determine ultimate strength, elongation, and yield strength. The latter property gives an excellent indication of the behavior of a wire under loads below the breaking strength. It indicates any change in strength before it might become detectable in grain size measurements and therefore is a valu able check on the weaving properties of the wire The short time necessary for computing the yield strength, also gives the test further advantage over the more lengthy metallographic procedure for determining grain size.

The modulus of elasticity, also, apparently has a marked effect upon the behavior of wires under the type of load experienced in the paper machine. A low modulus means lower stress values are required for any given elastic distortion. If the true proportional limit is too low, normal use of the wire may produce permanent deformation. Notable among the violators of this high modulus requirement were certain silicon-copper alloys which were tried for Fourdrinier wires. The material, literally stretched under normal loading conditions, until the Fourdrinier wire, although far from being worn out, was too long for the take-up rolls for the machine, and had to be scrapped.

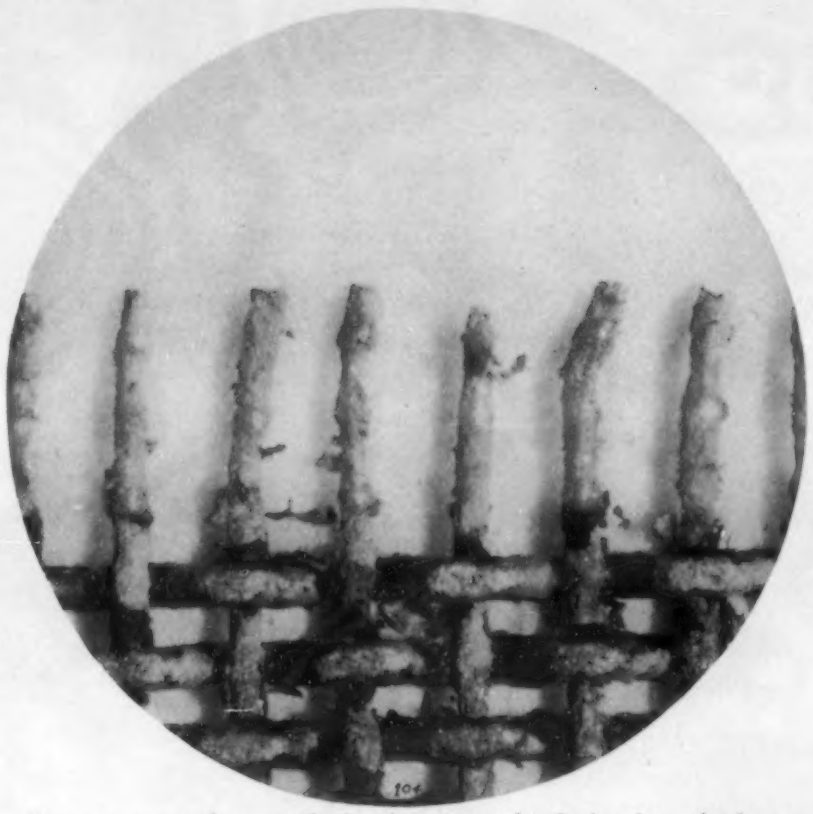
## Weaving

The longitudinal wires in the cloth, or the so-called "warp" wires are next wound on a large cylinder in the rear of the loom called the "beam roll." This is sectioned to take from 50 to 100 warp wires per groove, depending upon the mesh count of the wire being woven, so that there will be less danger of fouling of these wires as they are payed off into the loom to be woven into cloth. (With 2-in.

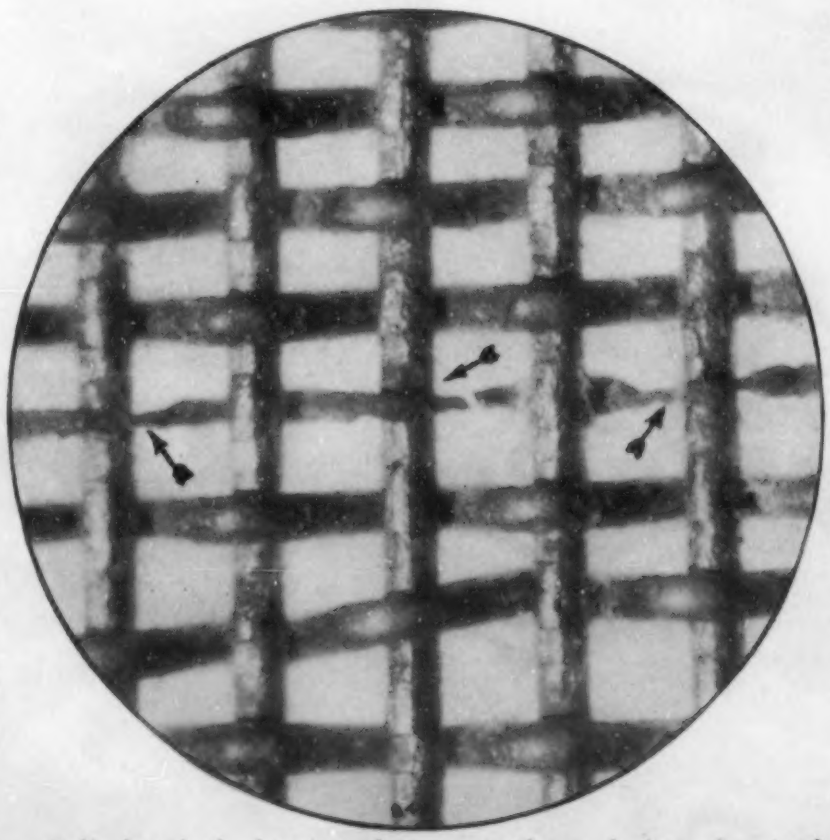
centers on the beam grooves, 25 mesh would contain 50 wires per groove; 100 mesh, 200 wires per groove). Each warp wire is as much as 4000 ft. long, joints being created by a special end-to-end brazing process with silver solder. As the spools of warp are prepared to be wound on the beam, they must be subjected to a uniform tension while the groove is being filled. If this were not accomplished, the cloth would contain longitudinal wrinkles because of non-uniform tension in the warp wires from one groove to another. The uniformity is obtained by "marshalling" (from the French to direct or guide) the wires over a series of bars, whereby each wire is brought to a uniform tension.

It is the length of the individual warp wires which is originally wound on the beam of a wire weaving loom which determines the number of Fourdrinier wires which can be made on the loom. Often, one loom is kept in operation by one, or a group of paper machines from one mill, using the same mesh count. This greatly facilitates prompt delivery. When special mesh counts are desired, frequently it is necessary to reserve a whole loom for this count, and if only one or two Fourdrinier Wires are ordered, the cost of such special work is far above average. If, however, the mill orders several of these special wires (The industry refers to the finished, seamed, and stretched screen cloth made for the Fourdrinier paper machine, as the "wire.") the cost will be very little higher than normal. The "winding on" period, in making up a loom, is the preliminary operation, which takes

*Wire weaving loom in action. The weaving face of the cloth is constantly under the inspection of the weaver. Special illumination at night prevents any imperfections in the surface characteristics.*



*Severe corrosion typical of waters high in free hydrogen sulphide. 25X.*



*Cylinder cloth showing almost complete solution of a single warp wire which touched the binding wire of a dissimilar metal (probably steel) producing marked galvanic corrosion. 30X.*

from two to three weeks.

When the beam is completely filled with warp wires, the free ends are brought in from the back of the loom to be attached to the cloth roll on the front of the loom on which the cloth will be taken up after weaving. Before reaching the cloth roll, they pass through the heddles, the means by which the warp wires are opened (alternately raised and lowered) to allow the shuttle to pass from one side of the loom to the other, paying off the shoot wire which has been wound on the shuttle bobbin. From the heddles the warps pass through the openings in the "reed." The reed is composed of many fine ribbons of hardened steel spaced between these warp wires. It is the reed, which is located at the weaving edge of the cloth, which "beats" the shoot wire into proper position across the warp. A special reed must be utilized for each "warp count" of wire being woven. The accuracy of the warp count of the wire depends upon the accuracy of the reed which positions the warp wires as the shoot wire is being driven into place.

The shoot wire is generally annealed to a softer temper than is the warp, for in the Fourdrinier, it is the warp which does the "work" of the cloth, the shoot being filler. The shoot or weft wire must be carefully wound on the shuttle bobbin before being inserted into the shuttle.

The shuttle bobbins on small looms hold from ½ to 1 lb. of wire. The bobbin is replaced at regular intervals with a full bobbin sometimes causing bands of color to appear across the cloth as it is woven. This color is caused by the tarnish on the shoot wire—varying from one spool to another. The spare ends are tied and broken off at the edge of the cloth where they do not interfere with the uniformity of the mesh openings. Cloth is woven at a rate of about 1 to 2 ft. per hr., depending upon the shoot count of the mesh being woven.

In the weaving operation many of the characteristics of the finished wire cloth are formed. The rigidity of the cloth, its resistance to curling, and the actual count of the mesh, all originate in the weaving operation. By varying the size of the shoot and warp wires, and their strength, numerous types of cloth may be produced for the production of various papers. Fourdrinier cloth is characterized by a shoot count which is practically always lower than the warp count. Thus news print is made on approximately 60 by 42 weave (60 warp wires and 42 shoot wires per inch of cloth), while some thin cigarette papers will require a mesh count of 70 by 210, using triple warp wires (grouped by three's).

When the proper length of cloth has been woven, the warp wires are cut, and the woven section is rolled off the cloth roll, onto the "cut-off roll" for transportation to the finishing operation.

The warp wires have conformed themselves closely

to the cloth roll in the front of the loom during weaving, and the cloth curls sharply in one direction. (The curl in the cloth is not actually caused by the warps being tightly wound over the cloth roll, but by the relative tension in the top and bottom sleds, or groups of wires manipulated by the heddles during weaving). This curl must be removed to facilitate further processing. This is accomplished by "springing" the length of woven cloth against its own curl by pulling the entire width over steel blades. In some cases a "springer" is incorporated in the loom itself. The cloth then lies flat on the cutting platform where it is cut to length for seaming. This length is always 1 or 2 ins. short of the Fourdrinier machine measurements. Following seaming the wire will be stretched to finished size, adding further strength and rigidity to the product, and reducing the amount of adjustment which might have to be made on the take-up rolls of the paper machine. *(To be concluded)*

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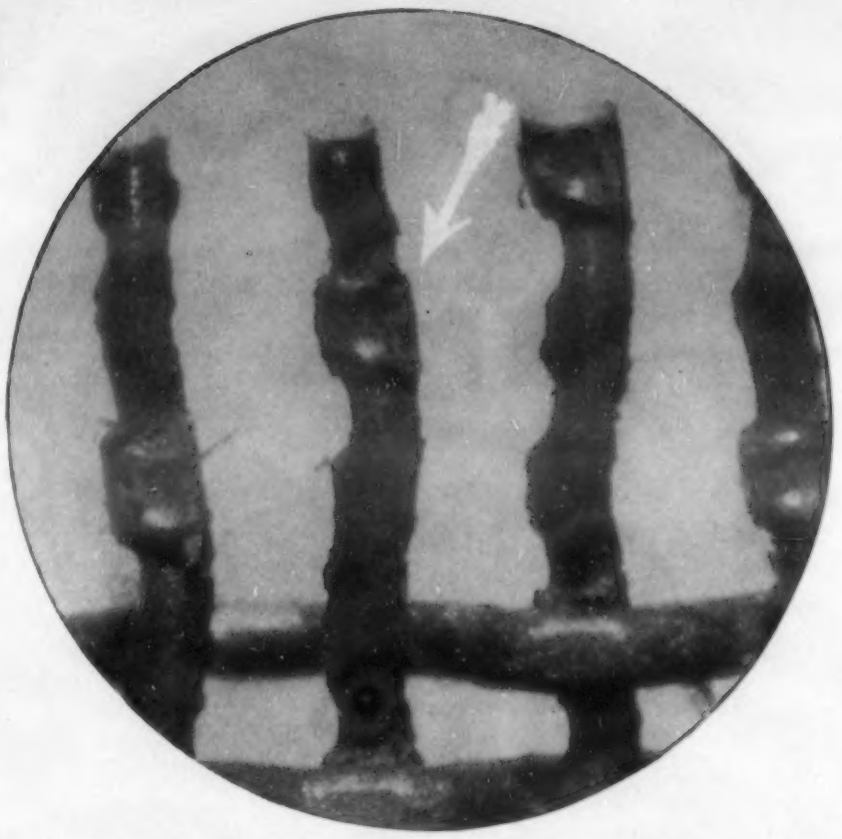
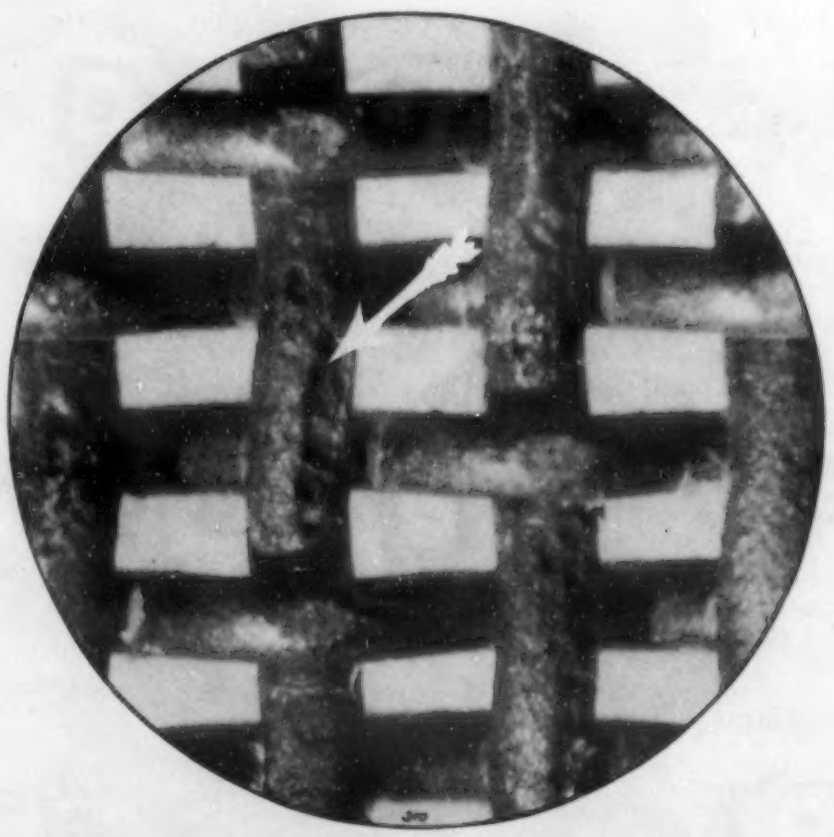


*Corrosion of brass shoot wire. Note how bronze warp wires at bottom of photo show very little corrosion. 40X.*



*Top side of Fourdrinier wire showing local corrosion on top side only, probably caused by a corrosive paper stock. 40X.*

# The Mercury Cathode in M

by A. E. PAVLISH and JOHN D. SULLIVAN

*Battelle Memorial Institute, Columbus, Ohio.*

*The development and widespread use of steels containing significant amounts of aluminum, vanadium, titanium, columbium and boron have focussed attention on the need for more rapid methods of determining these elements in the presence of large amounts of iron, nickel, chromium, etc. Complete separation by ordinary precipitation methods is difficult and tedious, but becomes simple, fast and practically foolproof when done electrolytically, using a mercury cathode. More general use of the mercury cathode has been retarded by lack of published information on the effects of kind and amount of free acid, and of iron concentration, on operation and results. This article reports on such effects and, in addition, suggests a simple method for recovering the mercury used.—The Editors.*

IN ACIDIC SOLUTIONS with a setup employing an insoluble anode and mercury as cathode certain elements are deposited in the latter whereas others are not. As a specific example, iron is deposited while aluminum is not; thus, the two can be separated completely and rapidly. The current and voltage requirements are such that a lead storage battery can be used if direct current is not available otherwise in the laboratory. A typical steel laboratory setup is shown in METALS AND ALLOYS, Vol. 9, July, 1938, p. 177.

Methods employing the mercury cathode rarely are used for the quantitative determination of elements deposited in the mercury because so many elements are deposited that this application of the method is limited. Removal of some elements from solution that interfere with the determination of other elements not deposited is readily effected. The mercury cathode can be classed as an extremely useful, but little used, tool in analytical chemistry.

*Table I. Elements Removed by Electrolysis with Mercury Cathode in Dilute (approximately 0.3 N) Sulphuric Acid Solution*[6]

| | |
|---|---|
| Elements quantitatively deposited in the mercury cathode | Cr, Fe, Co, Ni, Cu, Zn, Ga, Ge, Mo, Rh, Pd, Ag, Cd, In, Sn, Re, Ir, Pt, Au, Hg, Tl, Bi, Po |
| Elements quantitatively or partly separated from the electrolyte, but not quantitatively deposited in the mercury | Os, As, Se, Te, Pb, Mn, Ru, Sb |

The suggestion of precipitating metals in the form of amalgams was made in 1880 by Wolcott Gibbs.[1] Three years later Gibbs[2] again directed attention to the employment of the mercury cathode for the separation of iron, cobalt, nickel, zinc, cadmium and copper from solutions of their sulphates. The separation of interfering elements by electrolysis with the mercury cathode has been applied in the determination of aluminum,[3] boron,[4] vanadium,[5] etc. in carbon and alloy steels. This analytical tool is used in the laboratories of Battelle Memorial Institute for the separation of cobalt, nickel, iron, chromium and copper from aluminum and vanadium in steels and other alloys.

Table I shows the elements that are deposited or removed by electrolysis with the mercury cathode. The elements not shown in the table remain in solution under normal conditions.

Although use of the mercury cathode in analytical chemistry is old, the analytical chemist is handicapped in applying it to special problems because the effects of kinds of acids, varying concentrations of iron, and different degrees of acidity have not been described in the literature. One of the chief retardants to extended use of the method is the general impression that regeneration of the mercury is laborious, and that elaborate purifying equipment is necessary. The purpose of this article, therefore, is two-fold:

(1) To discuss the range of usefulness of the mercury cathode in analytical chemistry.

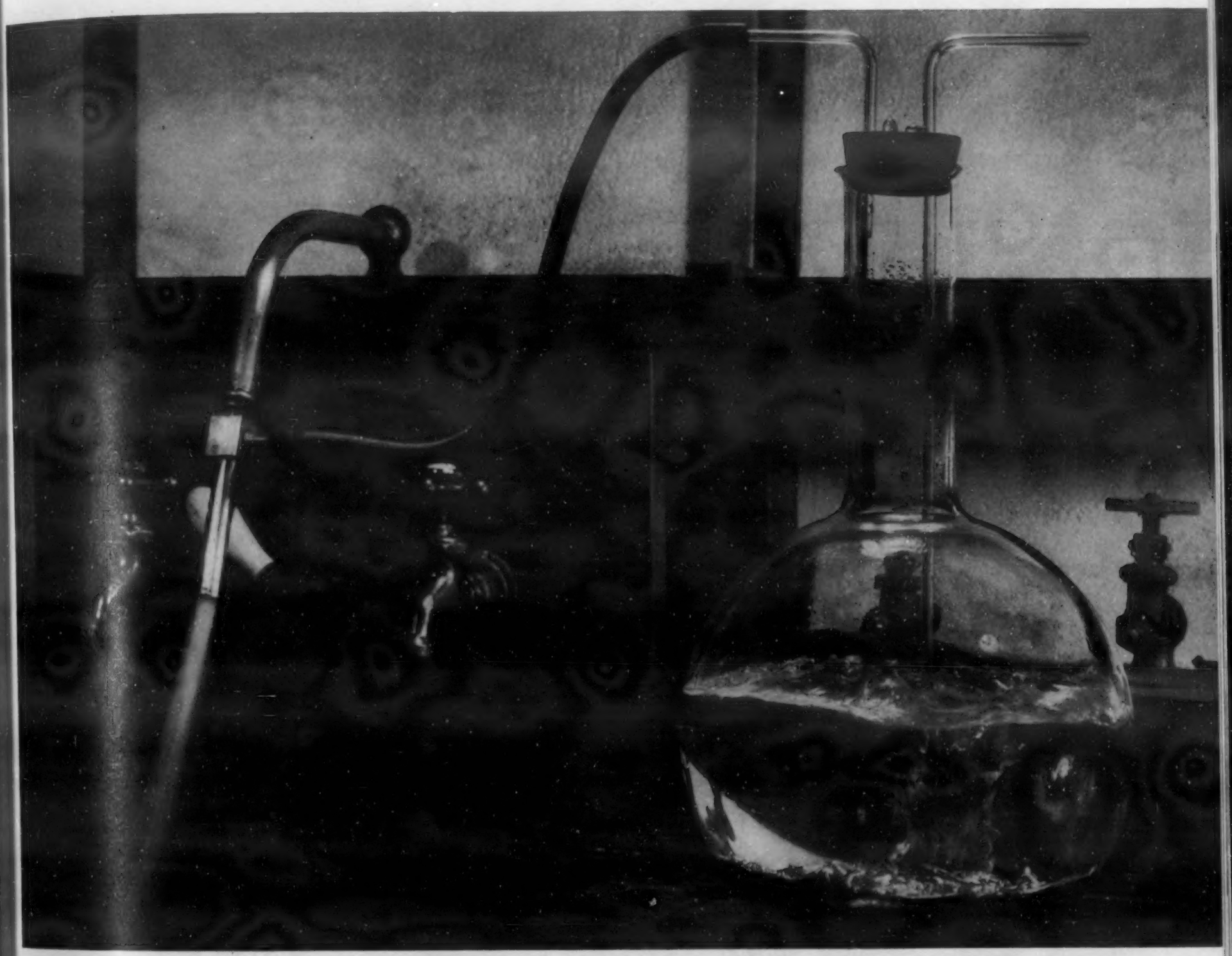
(2) To describe a simple method developed in this laboratory for purification of mercury.

The experiments were carried out in 400-ml. Pyrex beakers, using mercury as the cathode and sheet platinum as the anode. During electrolysis the contents of the mercury cell, including the mercury, were agitated with an electrically driven motor stirrer. The cell itself was immersed in a cooling bath maintained at 22 deg. C. The following conditions prevailed in the experiments:

| | |
|---|---|
| Volume of electrolyte | 150 ml. |
| Volume of mercury | 50 ml. |
| Anode area | 38.8 sq. cm. |
| Cathode area | 38.1 sq. cm. |
| Cathode current density | 0.13 amp. per sq. cm. |
| Voltage per cell | about 10 v. |

## Effect of Acidity

Two gm. of ingot iron were dissolved in such amounts of sulphuric acid that after dilution to the



*Regenerating mercury. Note simplicity of apparatus.*

desired volume the solution contained a definite concentration of free acid. The solution was electrolyzed, and deposition of iron was considered to be complete when a negative spot test was obtained on a drop of the electrolyte with potassium ferricyanide. The results are given in Table II. Data in the third column are average rates over the entire period of deposition. Instantaneous rates decrease as the iron becomes depleted. Deposition was effected even when the concentration of $H_2SO_4$ was as high as 10 *N* but the rate was somewhat lower at the high concentrations.

*Table II. Effect of Free Sulphuric Acia on the Rate of Deposition of Iron by Electrolysis with Mercury Cathode.*

| Free $H_2SO_4$, normality | Iron in electrolyte, grams | Rate of removal of iron, gm. per min. |
|---|---|---|
| 0.05 | 2.0 | 0.029 |
| 0.10 | 2.0 | 0.028 |
| 0.20 | 2.0 | 0.024 |
| 0.30 | 2.0 | 0.023 |
| 0.50 | 2.0 | 0.023 |
| 1.00 | 2.0 | 0.019 |
| 2.00 | 2.0 | 0.017 |
| 5.00 | 2.0 | 0.010 |
| 10.00 | 2.0 | 0.009 |

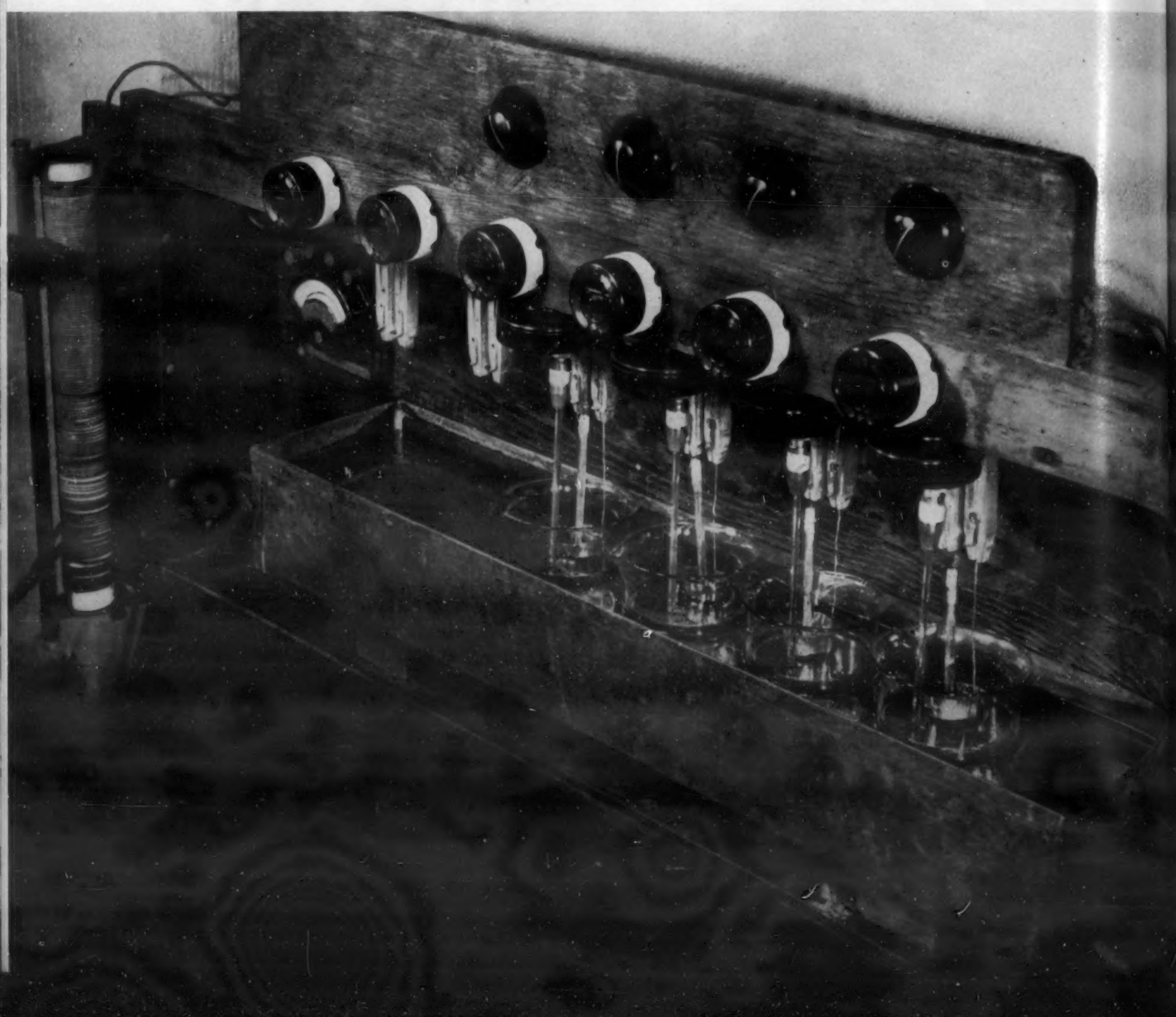
## Effect of Iron Concentration

To study the effect of concentration of iron in the solution on the rate of deposition and to determine the time required to deposit various amounts of iron, samples of ingot iron were prepared to give solutions of different concentrations of iron. In each instance the free $H_2SO_4$ was O.1 *N*. Results are given in Table III. The data in the fourth column show that the average rate of iron removal over the entire period was virtually the same when the concentration of iron was between 10 and 30 gm. per l. The overall rate of iron removal decreased rapidly when the concentration of iron was less than 5 gm. per l. Under the conditions of these experiments 5 gm. of iron can be deposited in the mercury in 3 hrs.

## Deposition from Various Acids

The literature ordinarily refers only to separations in sulphuric acid solutions. Modern analytical chemistry depends more and more on different reagents and combinations of acids. Perchloric acid, for example, is rapidly gaining in popularity and application. Experiments were made with the following solutions:

*Mercury cathode set-up at Battelle Memorial Institute.*



| Test | Iron in Electrolyte, gm. | Acid | Free acid, *N* | Rate of removal of Fe, gm. *per min.* |
|---|---|---|---|---|
| 1 | 2.0 | $H_2SO_4$ | 0.10 | 0.028 |
| 2 | 2.0 | $HClO_4$ | 0.10 | 0.028 |
| 3 | 2.0 | $H_3PO_4$ | 0.50 | 0.024 |
| 4 | 2.0 | $H_2SO_4$-$H_3PO_4$ | 0.30* | 0.025 |

* Sample dissolved in $H_2SO_4$ to give a final solution 0.1 *N* in free $H_2SO_4$; then $H_3PO_4$ was added in amount to give a solution 0.2 *N* in $PO_4'''$.

The data show that perchloric, phosphoric and mixed phosphoric-sulphuric acids are satisfactory for the removal of iron. The overall rates of iron removal from the various acids and mixtures were approximately the same as from sulphuric acid. Nitric and hydrochloric acids are not satisfactory for use in mercury cathode electrolysis.

## Recovery of Mercury from Iron Amalgam

As stated earlier, lack of knowledge of the ease of regeneration of mercury is a retardant to the extended use of the process. The authors recognized this, and investigated methods of simplifying the recovery. A description of the simple and inexpensive method that was developed follows:

About 15 to 20 lbs. of Fe amalgam to be cleaned are transferred to a heavy-walled Florence flask, covered with water, and agitated vigorously by means of a jet of air for one day or longer, depending on the iron content. The mercury is satisfactory for use in the mercury cathode when no further cloudiness of the water occurs on agitation with fresh water. If the laboratory is equipped with compressed air, this can be used as a source of agitation; otherwise use an ordinary laboratory suction pump attached to the water faucet. Substantially no mercury is consumed or lost in the determination or recovery.

*Table III. Effect of Concentration of Iron.*

| Sol'n. contained Fe, gm. per l. | Iron in 150 ml. used in test, gm. | Time required to remove all Fe, min. | Rate of removal of Fe, gm. per min. | Average current efficiency during deposition, per cent |
|---|---|---|---|---|
| 1.0 | 0.15 | 15 | 0.010 | 11.6 |
| 5.0 | 0.75 | 34 | 0.022 | 25.7 |
| 10.0 | 1.50 | 54 | 0.028 | 32.3 |
| 13.3 | 2.00 | 71 | 0.028 | 32.8 |
| 15.0 | 2.25 | 83 | 0.027 | 31.5 |
| 20.0 | 3.00 | 111 | 0.027 | 31.4 |
| 30.0 | 4.50 | 173 | 0.026 | 30.3 |

## Conclusions

For precipitation of iron in sulphuric acid solution, the normality of the acid may be as high as 10. The overall rate of iron removal is greatest when the concentration of iron is between 10 and 30 gm. per l. With a solution containing 1 gm. iron, electrolysis is complete in about 45 min.; with 5 gm., in about 3 hrs. Perchloric, phosphoric and phosphoric-sulphuric acids can be used as electrolytes. The simple air-agitation, water leaching method described is highly satisfactory for regeneration of the mercury.

*References*


[1] Wolcott Gibbs, New Item, *Chem. News,* Vol. 42, 1880, p. 291.

[2] Wolcott Gibbs, "Note on Electrolytic Determinations of Metals as Amalgams" (Refers to his talk in 1883), *Am. Chem. J.*, Vol. 13, 1891, p. 571.

[3] D. H. Brophy, "Aluminum in Alloys", *J. Ind. & Eng. Chem.*, Vol. 16, 1924, p. 963.

[4] H. A. Kar, "Determination of Boron in Steel", METALS AND ALLOYS, Vol. 9, July, 1938, p. 175.

[5] Lundell, Hoffman and Bright, "Analysis of Chrome-Vanadium Steel," *J. Ind. & Eng. Chem.*, Vol. 15, 1923, p. 1064.

[6] Lundell and Hoffman, "Outlines of Methods of Chemical Analysis", John Wiley & Sons, Inc., New York, 1938, p. 94.

# "18 and 8" and Related Stainless Steels

## Part II

by WALTER M. MITCHELL
*Metallurgical Engineer, Westfield, N. J.*

*Stainless steel panel Sheet hammered in relief.*

## Heat Treatment

The proper heat treatment of these steels is a most important factor in their usefulness. The Strauss patent applying particularly to the austenitic steels (U. S. Patent No. 1,404,908) specifies heating to 2000 to 2200 deg. F. and subsequent rapid cooling to produce a completely austenitic structure. Obviously, carbides should be in solution for maximum corrosion resisting qualities, but a scattered distribution of comparatively coarse carbide particles does not necessarily destroy usefulness. The very high temperatures specified by Strauss appear to be unnecessary and are, in fact, undesirable when the metal is to be used in ordinary applications where a polished surface will ultimately be required.

The carbon solubility curve for these steels, as found by Bain and Aborn, shows that for normal carbon content (0.10 per cent C) solution takes place beginning about 1750 deg. F. But when cooled from this moderate temperature the metal may still be too hard for easy fabrication and a somewhat higher temperature is generally found advisable. It is now customary to anneal or soften by heating in the range 1850 to 2000 deg. F., and cooling rapidly therefrom. For steels with lower chromium, such as the "Anka" type, the lower temperatures of this range will be sufficient; but for the high-chromium low-nickel type the higher temperatures will be necessary to produce maximum softness. When quenched, or cooled rapidly, with the method of cooling determined by the sectional thickness, these steels will consist entirely of austenite, unless the carbon content is high, in which case some free carbides may still be present, which are not necessarily harmful. It is not necessary to hold, or "soak," for a long time at the quenching temperature, unless the section be very heavy. In fact a long "soak" or too high a temperature is undesirable, for once the completely austenitic state is attained excess heat will merely coarsen grain structure and produce little further softening. Even though increased softness may be thus secured, its ad-

vantage is obtained at the cost of coarse grain structure, and this results in "orange peel" surface on sheet or strip products formed by bending or drawing. This is highly undesirable and seriously interferes with the production of a smooth polish on finished articles.

If the steel has been hardened by cold work, softening will begin at about 900 to 1000 deg. F., and continue progressively with higher temperatures. Softening seems to progress in two stages: one at about 900 to 1000 deg. F., the other at about 1550 to 1650 deg. F., and then continuously with decreasing effect at the highest temperatures. It seems probable that the retardation of softening in the range between 1000 and 1550 deg. F., is due to the precipitation of carbides, which is known to reduce ductility and stiffen the general structure of the steel. This retardation thus tends to counteract, in some measure, the softening that might be expected to be more directly proportional to the increase in temperature.

On account of the more sluggish nature of austenite the steels of this type do not develop the extremely coarse crystalline structure that is so characteristic of the ferritic straight chromium steels at higher temperatures. This is a distinct advantage, particularly in welding operations, for properly made welds remain ductile and require no grain refining treatment.

In contradistinction to general experience with ordinary carbon steels, coarse structure in chromium-nickel austenitic steels is not necessarily accompanied by brittleness or loss in ductility or toughness. Coarse-grained structure in castings is more or less inevitable, and while it may be refined in martensitic or pearlitic steels, such refinement is not possible with the austenitic castings. Although castings will show very coarse grain structure, they have a toughness and ductility that is somewhat surprising—fortunately for the foundries. Values of physicals for "18 and 8" castings after quenching from 1900 to 2000 deg. F., will average the following:

| | *0.07% C max.* | *0.10-0.20% C* |
|---|---|---|
| Tensile strength | 65-75,000 lbs. per sq. in. | 70-85,000 lbs. per sq. in. |
| Yield point | 30-40,000 " " " " | 35-45,000 " " " " |
| Elongation in 2 in. | 45-60% | 40-55% |
| Reduction in area | 45-60% | 40-55% |
| Bend | 180° | 180° |
| Brinell | 120-140 | 130-165 |
| Izod | 60-90 ft. lbs. | 60-90 ft. lbs. |

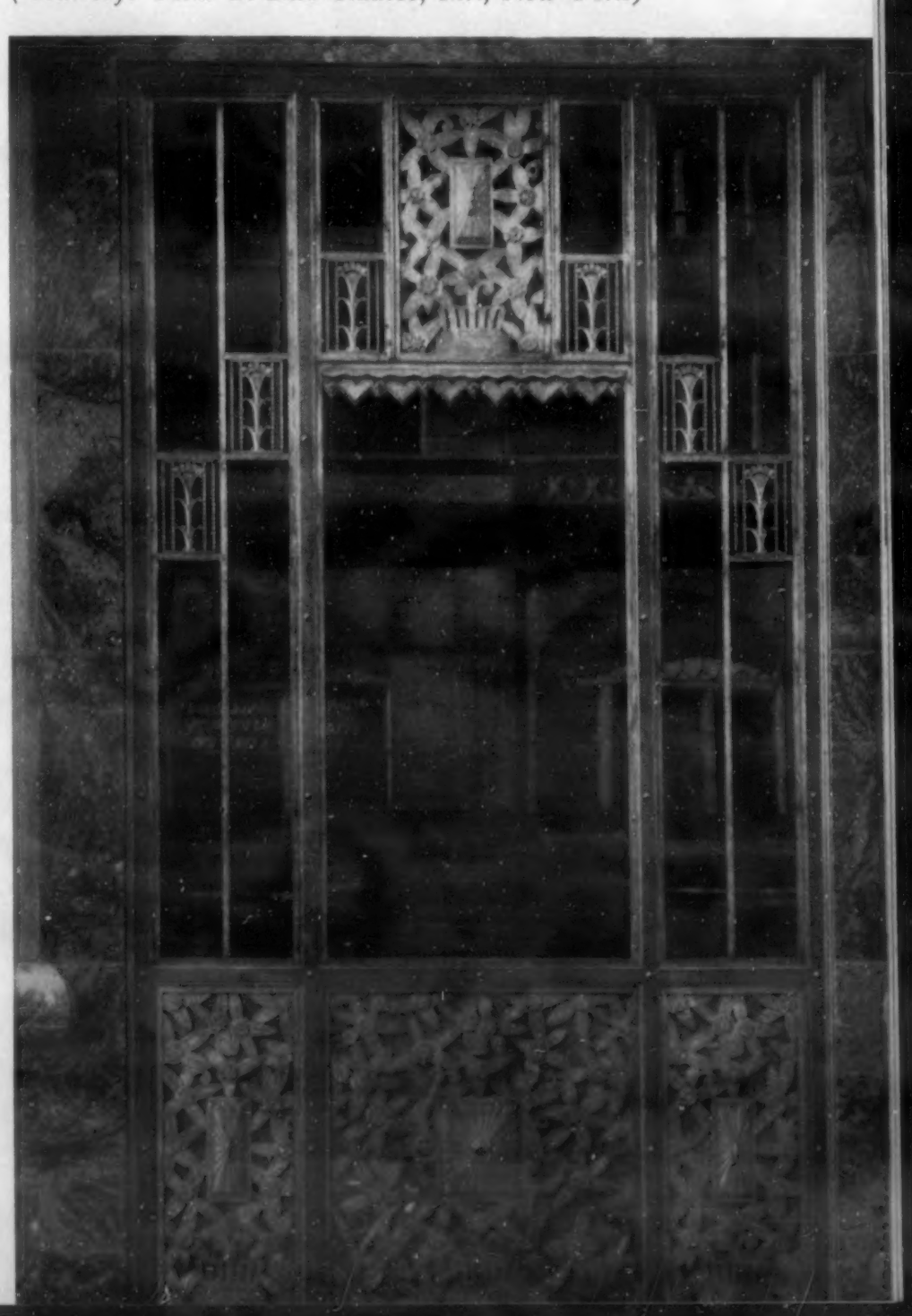
As has been remarked, since austenitic steels are without transformation ranges, grain refinement is impossible by any method of heat treatment alone, and may be accomplished only by mechanical working, either hot or cold. If by hot working, care must be observed that the subsequent annealing temperature is not excessive, otherwise the coarse structure will be re-established. The finest structure will be secured by severe cold working and subsequent rapid cooling from the range 1600 to 1700 deg. F. This treatment will create an entirely new crystalline structure, which will be very fine-grained, but the metal will be rather hard, with Brinell of 179 to 207. Quenching from a higher temperature will further soften the metal, but at the same time restore the usual polyhedral crystalline grains.

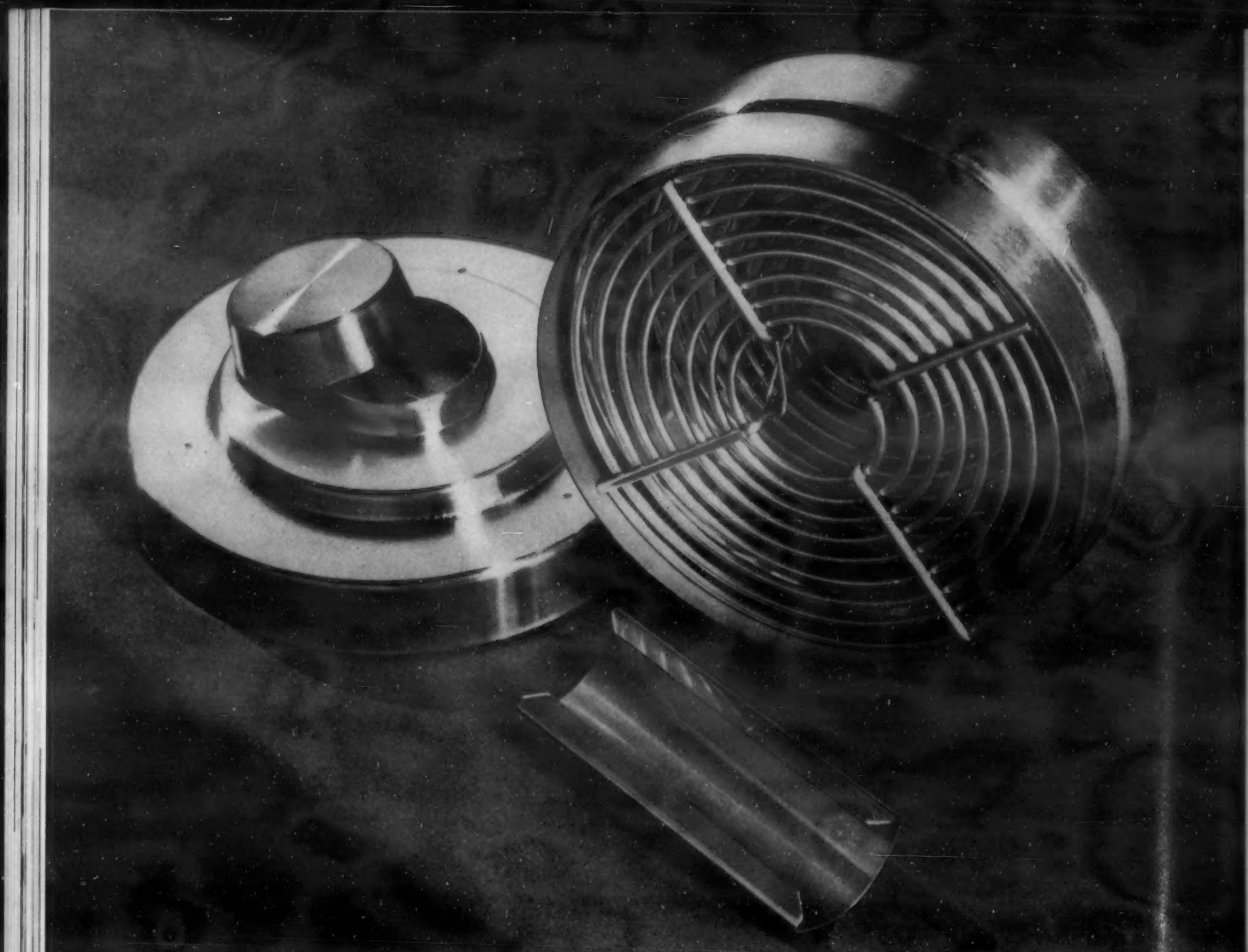
## Thermal Properties

All of the chromium-nickel austenitic steels have a high coefficient of thermal expansion. For the "18 and 8" analysis this is about 40 to 50 per cent greater than that of ordinary carbon steel in the lower temperature ranges. This high expansion must be carefully considered in the design of equipment that will be subjected to alternations of heating and cooling, and is highly important if other metals are to be included in the structure and rigid fastenings are employed in fabrication.

Furthermore, coefficient of expansion of the scale or oxide formed at high temperatures differs from

*Decorative window frame, stamped out of stainless steel, for Bell Telephone Co. building, Cincinnati. (Courtesy: Oscar B. Bach Studios, Inc., New York)*





*Reel and tank of "18 and 8" for developing small roll films.*

that of the metal itself. Thus, in cooling the scale will be thrown off, exposing the metal to fresh oxidation. This is true, especially, for the chromium-nickel steels and does not occur with the straight-chromium ferritic steels. It, however, restricts the use of the former for oxidation resistance where there are frequent alternations of heating and cooling.

The thermal conductivity of normal "18 and 8" is low, and is variously reported at values of 0.039-0.052 cal. per cub. cm. per sec. per deg. C., at 100 deg. C., increasing to 0.052-0.071 at 500 deg. C., and to 0.101 at 1000 deg. C.

While these values are low compared to other metals (about 1.00 for copper at 100 deg. C.) they are nevertheless higher, especially at elevated temperatures, than the values for the martensitic and ferritic stainless steels. The low thermal conductivity of "18 and 8" has been advanced as an objection to its use for various applications involving heat transfer, where its corrosion resistance would otherwise indicate it to be a most excellent material for the purpose. While the conductivity of the metal itself is important, the condition of its surfaces, i. e., the presence of scale or of deposits of foreign matter, and the "fluid films" on the surfaces of the metal in contact with gas or liquid, are very potent factors affecting heat transfer, and are exceedingly difficult to evaluate in mathematical terms. Where light gages are concerned these may have a greater effect on thermal efficiency than the conductivity of the metal itself. Much "18 and 8" has been used for equipment where good heat transfer characteristics are important, and no serious inconvenience because of its use has ever been reported to the author. Where heavy gages are concerned, however, the conductivity of the metal may well prove an adverse factor.

## Endurance Limit

So far the stainless steels have been employed chiefly because of corrosion resistance, but increasing

applications in engineering practice today make the subject of physical properties of various sorts of more than academic interest. One of these is "endurance limit." Investigations with various steels in the endeavor to find correlation between the endurance limit and other physical properties have established that endurance limit is apparently more closely related to the tensile strength than to any other physical property. This seems to be true for the austenitic stainless steels as well as for the martensitic carbon steels. Hence, for assurance of high endurance limit mechanical working or heat treatment should be such as will produce high tensile strength. In consequence, cold-worked material and relatively low annealing temperatures are to be preferred if this end is in view. For such material it has been found that the endurance limit will average better than 50 per cent of the tensile strength. In fully annealed, dead soft, material the endurance limit may be at a somewhat lower figure.

Data on endurance tests of "18 and 8" steels are scanty and unfortunately incomplete at best. Tests have been made by Oberg and Johnson at Wright Field with a rotating beam machine, and results on both notched and unnotched specimens are given. These may be summarized briefly:

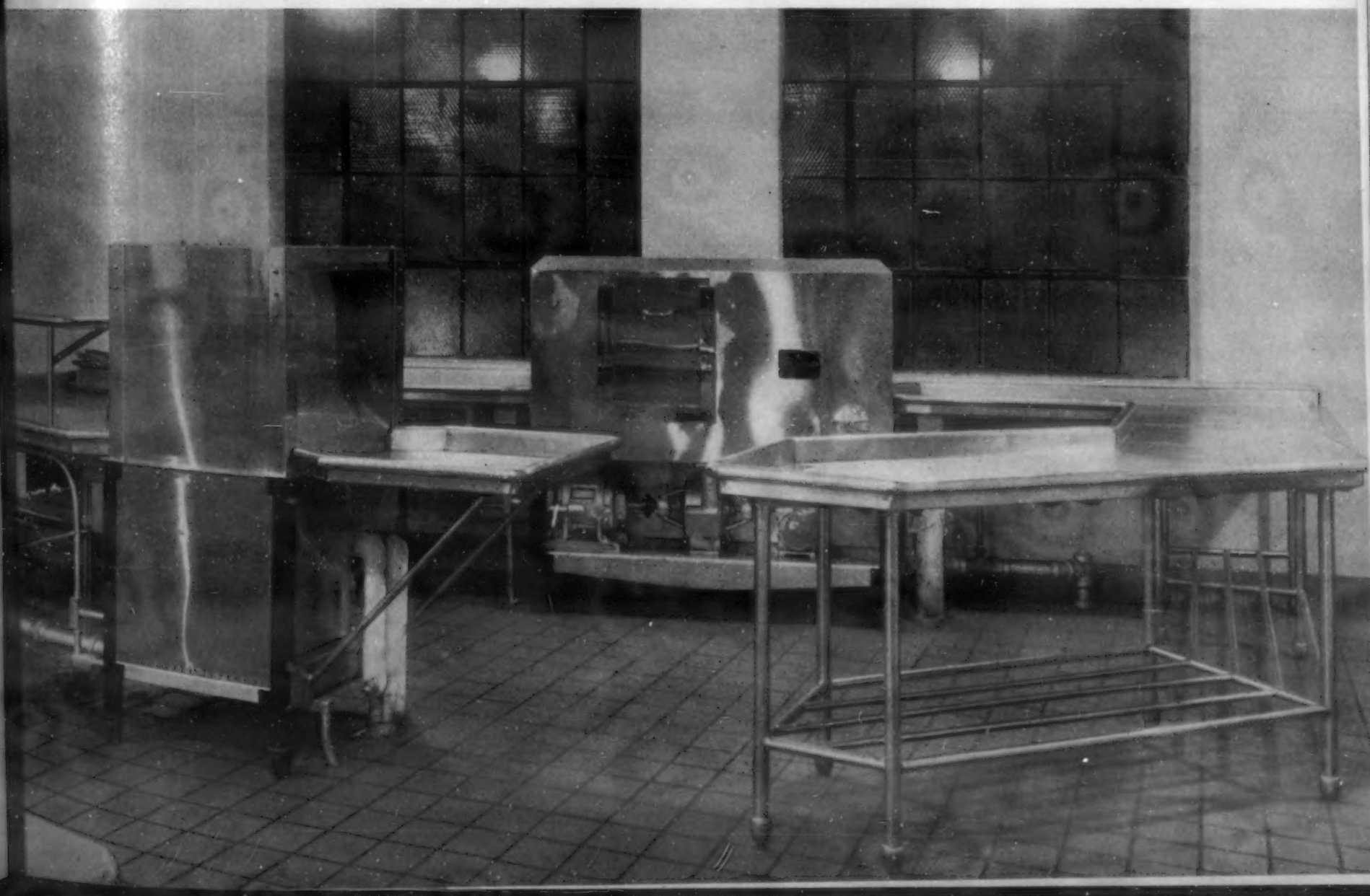
On annealed specimens of "20 and 10" (82,800 lbs. per sq. in. tensile strength) they find the endurance limit 51 per cent of tensile strength for unnotched, and 44 per cent for the notched specimens. For cold drawn "20 and 9" (132,500 lbs. per sq. in. tensile strength) they find the endurance limit 38 per cent of tensile strength for notched, and 53 per cent for the unnotched specimens. They remark that for annealed material, notched specimens show a higher endurance limit than unnotched, which curious phenomenon has been observed by others. With cold-drawn material, however, this is not the case, as this shows the usual higher value for the unnotched specimens.

Preliminary results of tests made by the National Tube Company several years ago to determine the endurance limit of normal "18 and 8" (maximum 0.08 per cent C) are given below. All specimens, except as noted, were drawn cold to smaller diameter and given various thermal treatments. Tests were made under completely reversed stresses on a Moore rotating beam machine.

| *Condition* | *Treatment* | *Tensile Strength lbs. per sq. in.* | *Endurance Limit lbs. per sq. in.* |
|---|---|---|---|
| Annealed | 9% reduction 1920 deg. F.-½ hr. water quench | 87,100 | 34,500 |
| Cold Drawn | 41½% reduction | 177,750 | 92,500 |
| Cold Rolled | As-rolled | 148,750 | 93,500 |
| Cold Drawn | 25½% reduction 1450 deg. F.-4 hrs. water quench | 93,650 | 72,000 |
| Cold Drawn (Stabilized-Ti) | 61% reduction 1525 deg. F.-3 hrs. air cool | 92,350 | 48,500 |

These results are averages, results on individual samples being somewhat erratic, and are to be considered as illustrative only.

*Stainless steel kitchen; installation at G. Fox Co., Hartford, Conn.*



As noted above, data on endurance tests of "18 and 8" are incomplete and scanty, and a more extended research program should be considered. Many factors are involved and careful research will be necessary to evaluate their individual effects.

## Fatigue and Stress Corrosion

It has been pointed out many times that the effect of corrosion simultaneous *with* stress is far more damaging than corrosion alone *followed* by stress. There are many factors involved and, where metals are concerned that have such selectively high corrosion resistance as "18 and 8" and its related steels, so much depends upon the condition of the metal itself and its surroundings that any prediction of life to be expected is certainly unsafe.

External stresses can usually be recognized and the possibility of their damaging effects may be guarded against, but the existence of internal stresses is not always appreciated, and may be an unsuspected reason for failure of the material.

A recent report by Hodge and Miller, of Babcock and Wilcox Co., giving the results of a study of failures of condenser and heat exchanger tubes extending over a period of years throws some interesting light on this subject. Their investigations indicate that internal stresses in austenitic stainless steels, although suspected, occur far more frequently than commonly supposed. They can remain as a sequel to fabricating operations, such as cold forming, welding, and even the rapid cooling recommended for assurance of an austenitic structure and solution of carbides, and are practically certain to occur in composite structures where austenitic and ferritic steels are joined by welding, notwithstanding subsequent careful slow cooling. Such steels, whether sensitized to intercrystalline attack (to be discussed in what follows) or not, are highly susceptible to stress corrosion from internal stresses, and this may result in failure by cracking, which may be intercrystalline or transcrystalline, or both, depending upon the existing conditions. And it is disturbing to note that this can occur when the stressed metal is exposed to certain chemical media that are ordinarily considered only mildly corrosive and do not attack carbon steel.

The remedy for such conditions, assuming that the metal is otherwise immune to attack from the corroding media, lies in the elimination, or reduction to a safe value, of any internal stresses, and this can be accomplished (except for complex structures for which there is apparently no remedy) only by relatively slow cooling, for which the use of "stabilized" alloys appears essential.

## High Temperature Properties

A discussion of high chromium-nickel alloys would be incomplete without some mention of the important properties at high temperatures imparted by the metal chromium itself. However, the subject is a very large one, and is so complicated and little understood that it demands separate consideration for its proper discussion, and brief mention only will be made here. The subject can be considered from two standpoints: Increased resistance to scaling, or oxidation, and maintenance of strength, or resistance to deformation under stress.

Considering first scaling, commonly published data show that "18 and 8" may be used with reasonable satisfaction at temperatures up to about 1600 to 1700 deg. F., for continuous service, and 1500 to 1600 deg. F., for intermittent service. The lower figure for intermittent service is the result of the tendency for scale to fly off during frequent cycles of heating and cooling through differential expansion, as already explained.

Obviously, the character of the atmosphere to which the metal is exposed will affect the actual results obtained—whether this is reducing or oxidizing, and what other gases may be present, especially those containing sulphur or water vapor (steam). These all influence the rate at which scaling may take place; so that, as with laboratory tests of corrosion resistance, any experiments to estimate probable life of equipment will be only approximate, unless exact conditions of service can be duplicated. Not only will the constitution of the atmosphere affect the results, but, naturally, variations in the composition of the metal will affect these also, as will be noted in what follows. Increased chromium raises the limits of scaling temperature ranges. Nickel is not so effective in this respect unless the percentage is materially increased (20 per cent and upwards), when it definitely increases scaling resistance.

The maintenance of strength at high temperatures —creep strength—is too complex a subject, and still too little understood for its discussion within the limits of this article. *(To be continued)*